JACQUES, WHITFORD & ASSOCIATES LIMITED

CONSULTING ENGINEERS

CAPE BRETON COAL RESEARCH LABORATORY

STABILITY OF COAL MEASURES STRATA WITH WATER-BASED CHEMICAL AGENTS (PART 2)

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STABILIZATION OF COAL MEASURES STRATA WITH WATER-BASED CHEMICAL AGENTS - PART 2

BY

JACQUES, WHITFORD & ASSOCIATES LIMITED

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EXECUTIVE SUMMARY

This report describes laboratory testing performed on seatearth samples taken from three coal workings in Cape Breton: the Lingan 6W-TOP, the Lingan 9E-TOP, and the Evans Mine, together with a sample of Bearpaw Shale from Saskatchewan. The objectives of the testing were to: (a) experiment with laboratory procedures which would identify and classify problem seatearths which may degrade or soften with detrimental effects to the workings; and (b) to experiment with water-based chemical agents which would inhibit degradation.

The Lingan samples were classified as non-swelling, non-softening and non-degrading. The Evans sample exhibited mechanical degradation under an (accelerated) ultrasonic bath dispersion test and under slake durability testing, attributed mainly to microfracturing in the rock structure.

The Bearpaw Shale exhibited true particle dispersion and softening under the test procedures, and exhibited irreversible changes in properties when dried.

Dispersion of the Evans shale and Bearpaw Shale was reduced when various combinations of mineral salts and polymers were added to the immersion water. The most effective treatment was the addition of 7.9 percent (dry weight) of potassium chloride (KCl), Polymers, while equally effective in combination with smaller amount of KCl, produce slimy liquids and slippery surfaces and are not suited to a mine application.

SOMMAIRE EXECUTIF

Cet étude englobe les essais en laboratoire exécutés sur des échantillons de semelles de mine obtenues auprès de trois chantiers d'exploitation du charbon au Cape Breton, notamment le Lingan 6W-TOP, le Lingan 9E-TOP et la mine Evans en plus d'un échantillon de schiste argileux de Bearpaw en Saskatchewan. Ces essais avaient pour but d'identifier et de classer les semelles de mine problématiques à l'aide de techniques de laboratoire ainsi de faire des expériences sur des agents chimiques à base d'eau qui entravent la dégradation. Les semelles de mine problématiques peuvent se dégrader ou s'assouplir occasionnant des effets nuisibles au chantier d'exploitation.

On a classé les échantillons obtenus à Lingan en tant que non-gonflé, non-adoucissant et non-dégradant. L'échantillon de Evans a démontré une dégradation mécanique pendant un essai de dispersion par bain ultrasonore (accéléré) et durant un essai de résistance à l'extinction. Cette dégradation peut être attribuable en grande partie à la microformation de fissures à l'intérieur de la structure de la roche.

Le shiste argileux de Bearpaw démontra une dispersion de particules véritable et adoucissement durant l'essai de techniques. Cet échantillon a également présenté des changements de propriétés irréversibles une fois desséché.

L'étude a également démontré que l'effet des dispersions des schistes argileux de Evans et de Bearpaw étaient réduits quand divers mélanges de sels minéraux et de polymères furent ajoutés à un eau de dissolution. Le traitement le plus efficace comprenait l'addition de 7.9 pourcent (poids pour matières sèches) de chlorure de potassium (KCl). Les polymères, quoique aussi efficaces en combinaison avec des plus petites quantités de KCl, produisent des liquides boueux et des surfaces glissantes qui ne sont pas convenables pour l'usage dans les mines.

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1.0 INTRODUCTION

1.1 Scope of Work

Seatearths, shale strata which typically lie stratigraphically below the coal seam, often present problems of stability to underground coal mine workings. The strength of the seatearths are known to be diminished by the adsorption of water from various sources such as the mine environment and connate waters. The adsorption of water of a shale is dependent upon a number of factors, among them the clay mineralogy, fractures, and permeability. Other workers particularly in oil well drilling applications, have found a number of aqueous based chemicals which inhibit the adsorption of water and increases stabilization of shale strata.

The Cape Breton Coal Research Laboratory (CBCRL) commissioned Jacques, Whitford & Associates Limited (JWA) to classify and profile problem and non-problem seatearths, as well as investigate the effect of aqueous-based chemical agents on the degradation of problem seatearths.

1.2 Conduct of Study

Seatearth samples from Lingan and Evans mines were supplied to Jacques, Whitford and Associates by the Cape Breton Coal Research Laboratory. These samples were classified based on procedures outlined in the statement of work attached in Appendix A-1. The results of these tests clearly indicated lack of swelling clays present in these shales, and suggested that the study would not be fruitful. However, the program was continued since the Evans shale exhibited some mechanical dispersion and breakdown. To obtain a clearer understanding of swelling clays and their behaviour, a sample of Saskatchewan Bearpaw Shale was acquired and subjected to classification and profiling as outlined by Appendix A-1. Because the number of samples, and the number of problem shales were greatly reduced, the scope and cost of the program were also reduced.

To investigate the effect of aqueous-based chemical agents on the degradation of problem shales, a test procedure was developed which utilized an ultrasonic bath into which problem shales were immersed. This procedure had not been part of the original "Statement of Work," but its use was accepted.

This report is presented in five sections: Section 1 deals with the conduct of study task, Section 2 describes the methods employed, and Sections 3 and 4 present and discuss the results of the work undertaken. Section 5 presents conclusions drawn from the results of the tests.

2.0 LABORATORY TESTING PROGRAM

2.1 Classification of Seatearths, Testing Procedure

The shale samples received in the laboratory were classified based on the following:

- water content;
- porosity and density;
- Atterberg Limits;
- slake durability; and
- grain size.

The procedures are outlined briefly below. Full details are given in Appendix C.

Water Content

Water content determination was executed on both intact and crushed samples. The intact samples had an approximate volume of 500 cm^3. The crushed material was sieved on a #10 mesh. All material passing through the No. 10 mesh was used for crushed moisture content. The moisture contents were determined as outlined in the Pit Slope Manual, CANMET Report 77-25.

Porosity and Density

Sample preparation and testing procedure were both executed in accordance with procedures described in the Pit Slope Manual, CANMET Report 77-25.

Atterberg Limits

Sample preparations and methods for determining the Atterberg Limits were tested according to the following procedures: ASTM D423 - Liquid Limit of Soils; and ASTM D424 - Plastic Limit of Soils.

Grain Size Distribution

The sample was ground, put through a #10 sieve, and was soaked for 16 hours in Calgon before the testing started.

The values of specific gravity used in the calculation of the hydrometer results were taken from the Porosity/ Density tests.

All samples were tested by Hydrometer and were executed in accordance with ASTM procedure D422 - Particle Size Analysis of Soils.

Slake Durability Index

The sample preparations and testing procedure were both conducted in accordance with procedures outlined in the Pit Slope Manual, CANMET Report 77-25.

2.2 Profile on Seatearth Samples

To profile both problem and non-problem seatearths, the following tests were conducted:

- X-ray diffraction
- adsorption isotherm
- sonic dispersion test

X-Ray Diffraction

The X-ray analyses of the Seatearth samples was sub-contracted to Cuesta Research Ltd., affiliated with Dalhousie University. The samples were ground and examined in an X-ray diffractometer. The diffracted energy was recorded on a chart recorder. An interpretation of the mineralogy was made by calculating the D-spacing using Bragg's equation. A more detailed explanation of the procedure and results are contained in Appendix C-7.

Adsorption Isotherm

The adsorption isotherms (A.I.) of problem and non-problem seatearths were determined from a procedure outlined by Chenevert[1], (October 1970), Appendix C-8. The A.I. relates the activity of the shale to its water content.

The adsorption characteristics of ground seatearth material less than No. 10 mesh size was determined for each sample. The adsorption characteristics of ground Evans and Bearpaw samples were determined after being soaked for 24 hours in 7.9 percent potassium chloride solution and then direct to a constant weight. It should be noted that to avoid two drying stages, the samples were ground at their original moisture content, producing a fraction greater than No. 10 mesh.

The A.I.'s were obtained by placing dry minus No. 10 mesh material in desiccators having controlled activity atmospheres. The samples were weighed daily until they were in equilibrium with the atmosphere in the desiccator. The final sample weights were then used to calculate the percent by weight of water adsorbed.

Sonic Dispersion Test

Dispersion refers to continuous and often rapid disintegration of the shale surface. An accelerated dispersion test was developed in the Jacques, Whitford and Associates laboratory by placing the seatearth samples in water or in an aqueous based solution and subjecting the samples to ultrasonic energy. Seatearth samples consisting of one intact lump of 50-100 grams weight at in situ moisture condition were immersed in a Bransonic ultrasonic bath for 2 hours. Samples were then removed from the bath and washed on a No. 200 mesh seive, dried, and sieved so that the dispersion characteristics could be described in terms of changes in particle size gradation.

2.3 Aqueous Based Chemical Agents and Simulated Degradation of Problem Shales

Seatearth samples exhibit varying rates of degradation depending on the aqueous based chemicals in solution. The following chemicals and concentrations of chemicals were tested for their effect on the degradation of the seatearth samples. Using the dispersion test as outlined in Section 2.2 accelerated dispersion or degradation of seatearth samples could be quickly observed.

Sodium Chloride	5.4%	NaCl
	7.9%	NaCl
Potassium Chloride	5.4%	KCl
	7.9%	KCl
Calcium Chloride	4.76%	$CaCl_2$

	General Name	Brand Name	
Polymers	Polyacrylamides	Alcomer[a]	with 7.9% KCl
			5.4% KCl
			3.0% KCl
	Polyacrylamides	SS-100[b]	with 7.9% KCl
			5.4% KCl
			3.0% KCl

(a) supplied by Dresser Magcobar

(b) supplied by Technifluids.

3.0 RESULTS OF LABORATORY TESTS

Results of the tests are presented herein. Complete laboratory data are included in Appendix B.

3.1 Results of Classification Testing

Water Content

The water content of each of the four samples are as follows:

	Evans Mine	Lingan Mine 6W-Top	Lingan Mine 9E-Top	Saskatchewan Bearpaw Shale
Samples (intact)	3.37%	1.23%	0.66%	25.76
Samples (crushed)	3.71%	0.76%	0.61%	23.25

Porosity and Density

Complete results of the porosity and density tests are shown on Figures 19 to 22, Appendix B. The samples have been tested for density using five or more 40-100 gram specimens. The dry density, the grain density, porosity and degree of saturation were determined on each specimen. These values were averaged and the results may be summarized as follows:

	Evans Mine	Lingan Mine 6W-Top	Lingan Mine 9E-Top	Saskatchewan Bearpaw Shale
Dry Density d(g/cm^3)	2.45	2.65	2.75	1.85
Grain Density g(g/cm^3)	2.55	2.65	2.75	2.71
Porosity, n	3.9%	0.0%	0.0%	31.7
Degree of Saturation, Sr	100.0%	100.0%	100.0%	-

It is noted that test results for the grain density, G_s have been corrected for minor inaccuracies during the testing procedure.

Values of dry density and grain density for the Evans Mine are lower than for most rocks and minerals.

Bearpaw Shale has a very high porosity and moisture content which yields a very low dry density. The measured value of degree of saturation has been affected by shrinkage of the samples during oven drying, and hence is not shown on the above Table.

Atterberg Limits

The Atterberg Limits on the sample of the Evans Mine resulted in a low plasticity index (Ip=8). The plastic limit and the liquid limit have values of 20.2 and 20.8 respectively. The samples of the Lingan Mine were non-plastic.

The Atterberg Limits on the Saskatchewan Bearpaw Shale resulted in a high plasticity index (Ip = 48.6). The plastic limit and liquid limit have values of 29.1 and 77.7 respectively. Figure 1 illustrates the plasticity of the samples.

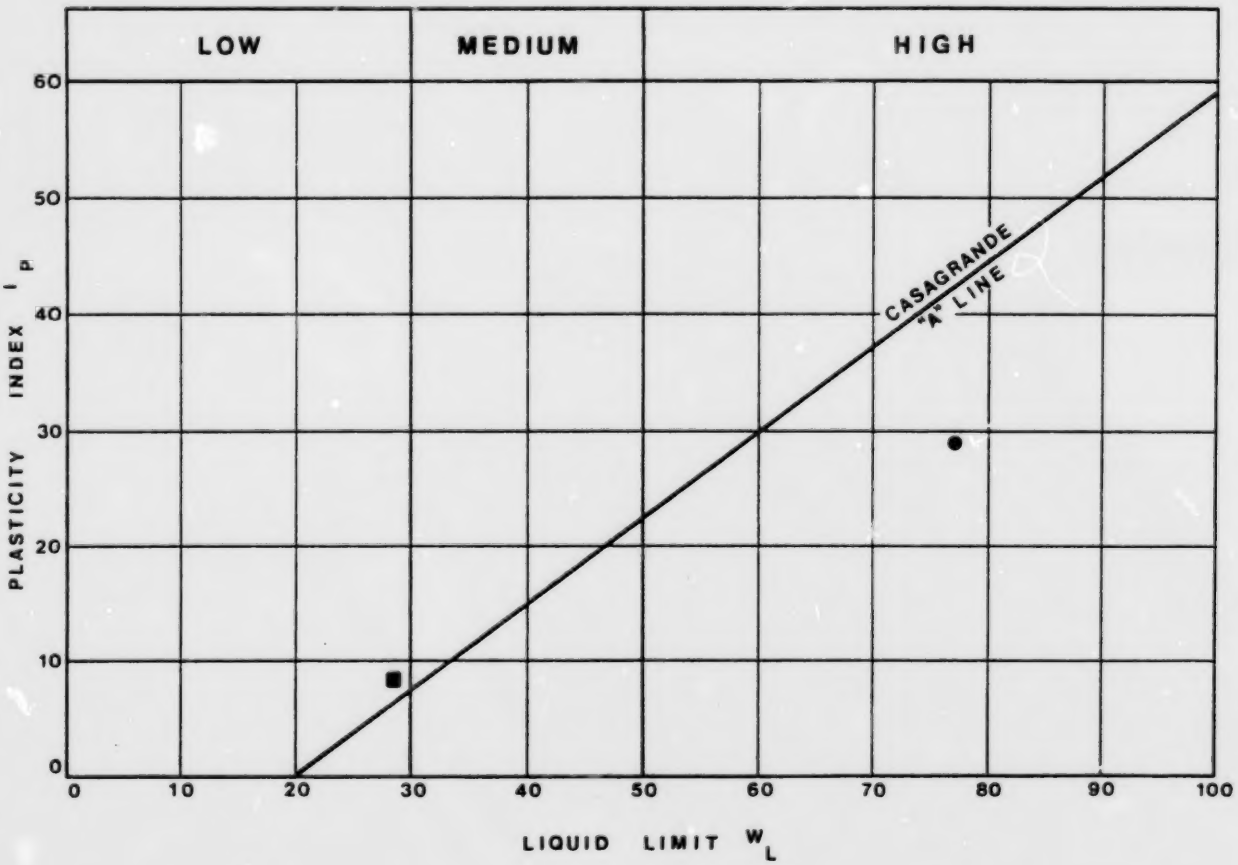
Grain Size Distribution

Grain size analysis on the four samples was conducted by hydrometer. The results of the hydrometer tests are shown on Figures 2 and 3.

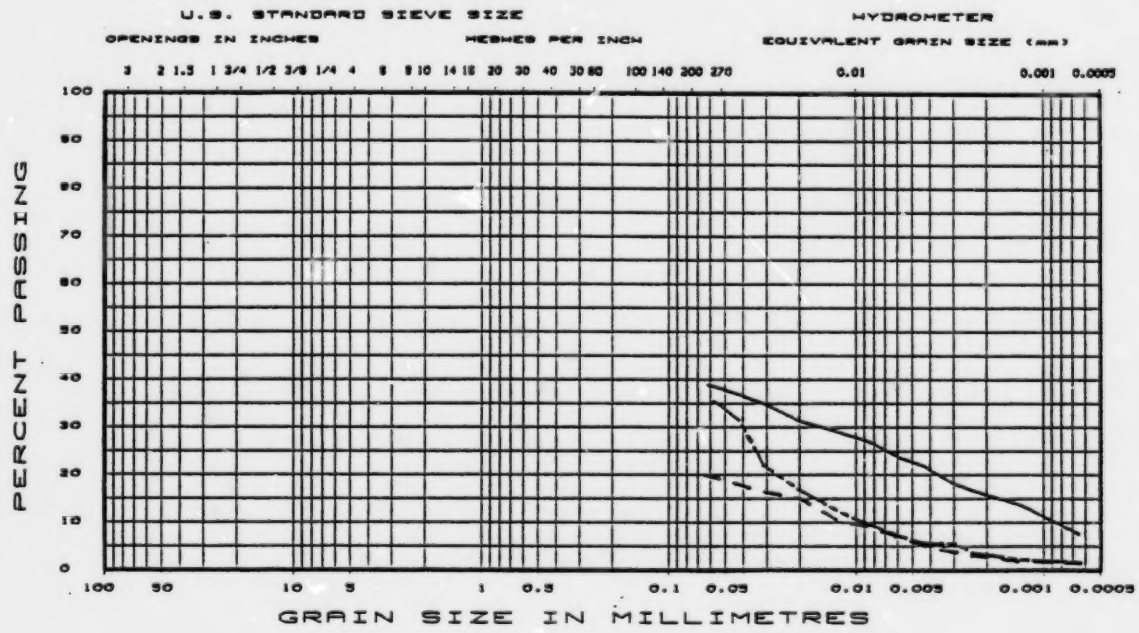
Slake Durability

The slake-durability index for the Evans Mine, Lingan Mine 6W-Top and Lingan Mine 9E-Top were 62%, 94% and 98% respectively. The Bearpaw Shale had a slake-durability index of 19.5%. The results of the slake tests are shown in Figure 17.

PLASTICITY CHART



SYMBOL	SAMPLE
■	EVANS MINE
●	BEARPAW SHALE

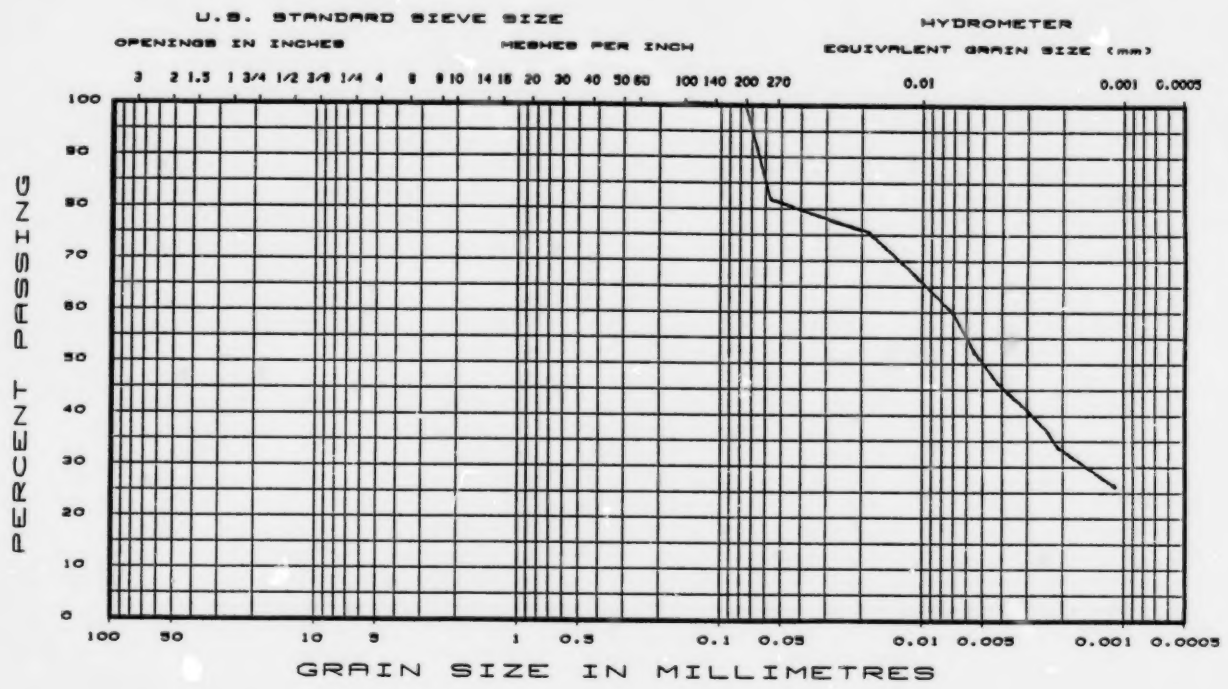

U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 8 10 14 16 20 30 40 50 80 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

——— EVANS MINE
— — — LINGAN MINE 6W-TOP
———— LINGAN MINE 9E-TOP

GRAIN SIZE GRAPH OF EVANS,
LINGAN 6W-TOP & LINGAN 9E-TOP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0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

DESCRIPTION
BEARPAW SHALE

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
.1490	0.0	0.0	0.0	0.0	100.00
.0740	.2	0.0	0.0	0.0	99.68
.0537	0.0	.5	19.5	47.0	81.46
.0398	0.0	1.0	19.5	46.0	79.46
.0284	0.0	2.0	19.5	45.0	77.46
.0182	0.0	5.0	19.5	43.8	75.06
.0109	0.0	15.0	19.5	39.8	67.06
.0069	0.0	39.0	20.0	33.8	59.48
.0054	0.0	68.0	20.0	32.0	51.88
.0042	0.0	120.0	20.0	29.0	45.88
.0029	0.0	240.0	22.0	25.5	40.56
.0024	0.0	380.0	23.0	23.0	36.40
.0021	0.0	490.0	23.5	21.2	33.22
.0011	0.0	1965.0	22.0	18.0	25.56

Sample weight for sieve analysis = 50.0 grams.
Sample weight for hydrometer analysis = 50.00 grams.
Specific gravity used in hydrometer analysis = 2.69

GRAIN SIZE GRAPH AND DATA OF BEARPAW SHALE

The general appearance of fragments of samples from the Evans Mine retained in the drum after testing may be described as thoroughly liberated angular fragments. The original ten rock lumps were broken down into approximately 100 to 120 smaller pieces. Approximately 30% of the material that passed through the drum was retained on the #200 sieve.

For both samples of the Lingan Mine, the originally tested rock lumps were somewhat rounded but not broken. The material that had passed through the drum consisted mostly of very fine particles.

The Bearpaw Shale samples were greatly reduced in size, but did not break up like the Evans shale. Fines were liberated creating a sludge inside the reservoir.

3.2 Results of Profiles on Seatearth Samples

X-Ray Diffraction

The X-ray diffractograms of the seatearth samples are shown in Figures 28 to 31, Appendix B, and reveal the following:

The two samples from Lingan, (Lingan 9E and Lingan 6W) are similar. The samples contain: illite and kaolinite clays; silt size grains consisting of quartz; and iron rich chlorite may be present.

The Evans sample contains clays consisting predominantly of illite and kaolinite. This sample also contains a high percentage of quartz. There is no evidence of chlorite; however, there is some evidence of pyrite.

The Bearpaw Shales contains clay and quartz. The clay minerals have been identified as montmorillonite, illite and kaolinite.

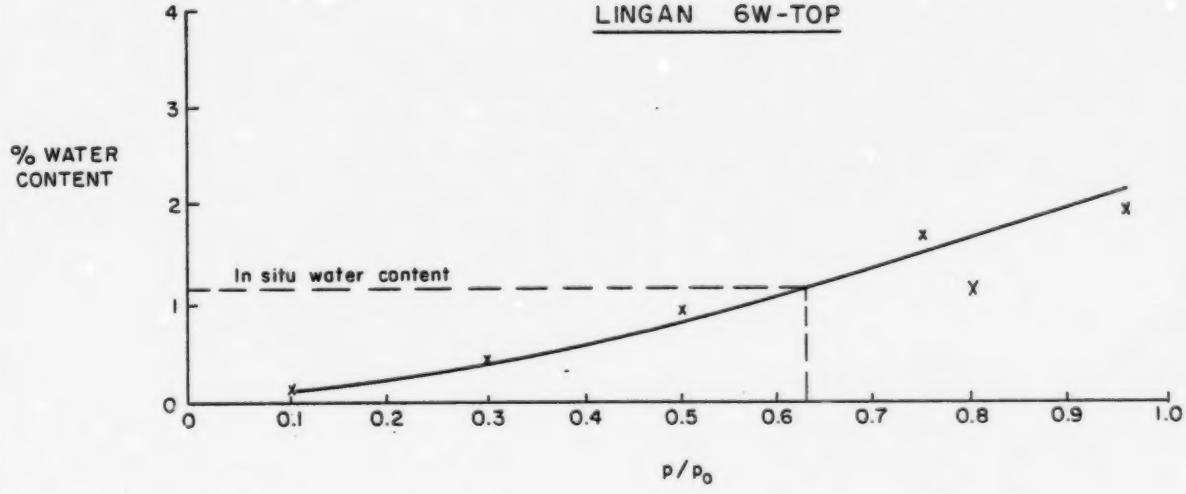
Adsorption Isotherm

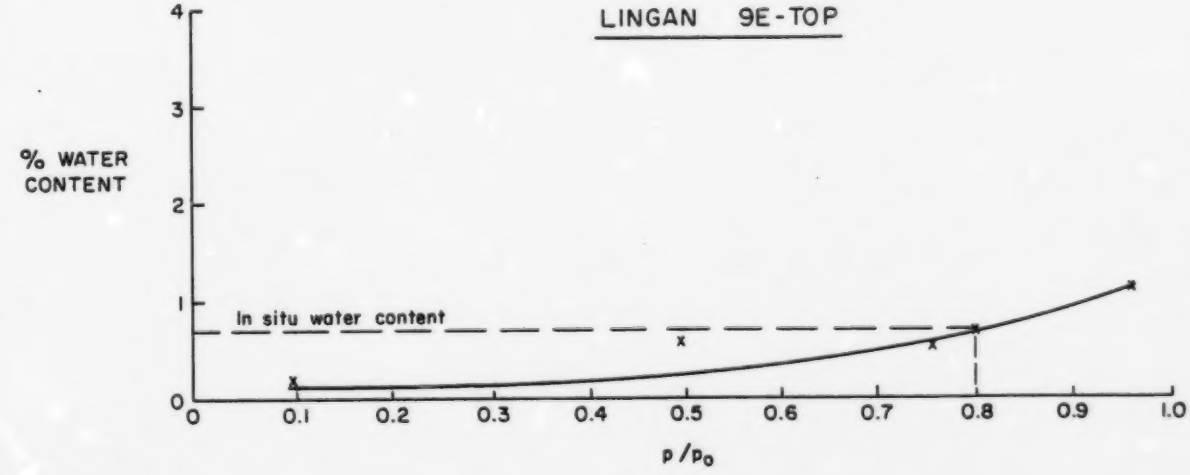
The results of the adsorption isotherm tests for the four samples are shown in Figures 4 to 8.

ADSORPTION ISOTHERM TESTS (UNTREATED)

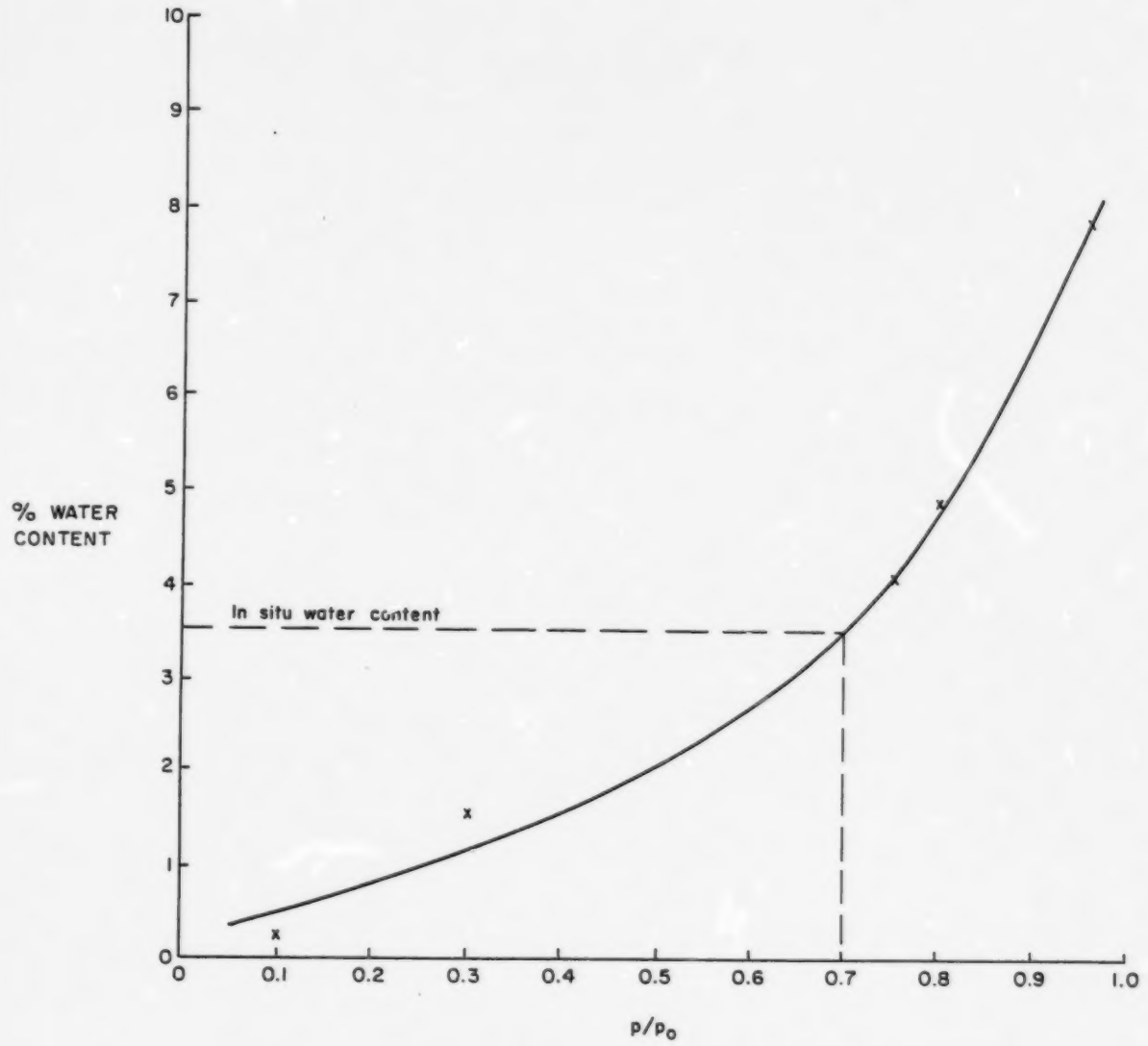
Salt Employed	p/po	Moisture Content Lingan 9E	Lingan 6W	Evans	Bearpaw
$ZNCl_2$	0.100	0.19	0.13	0.28	0.35
$CaCl_2$	0.295		0.40	1.56	1.90
$Ca(NO_3)_2$	0.505	0.6	0.9	3.9	4.2
NaCl	0.755	0.5	1.59	4.06	5.08
$(NH_4)_2SO_4$	0.800	0.66	1.17	4.9	5.8
KH_2PO_4	0.960	1.12	1.87	7.9	9.25

p/p_o = water activity or relative humidity

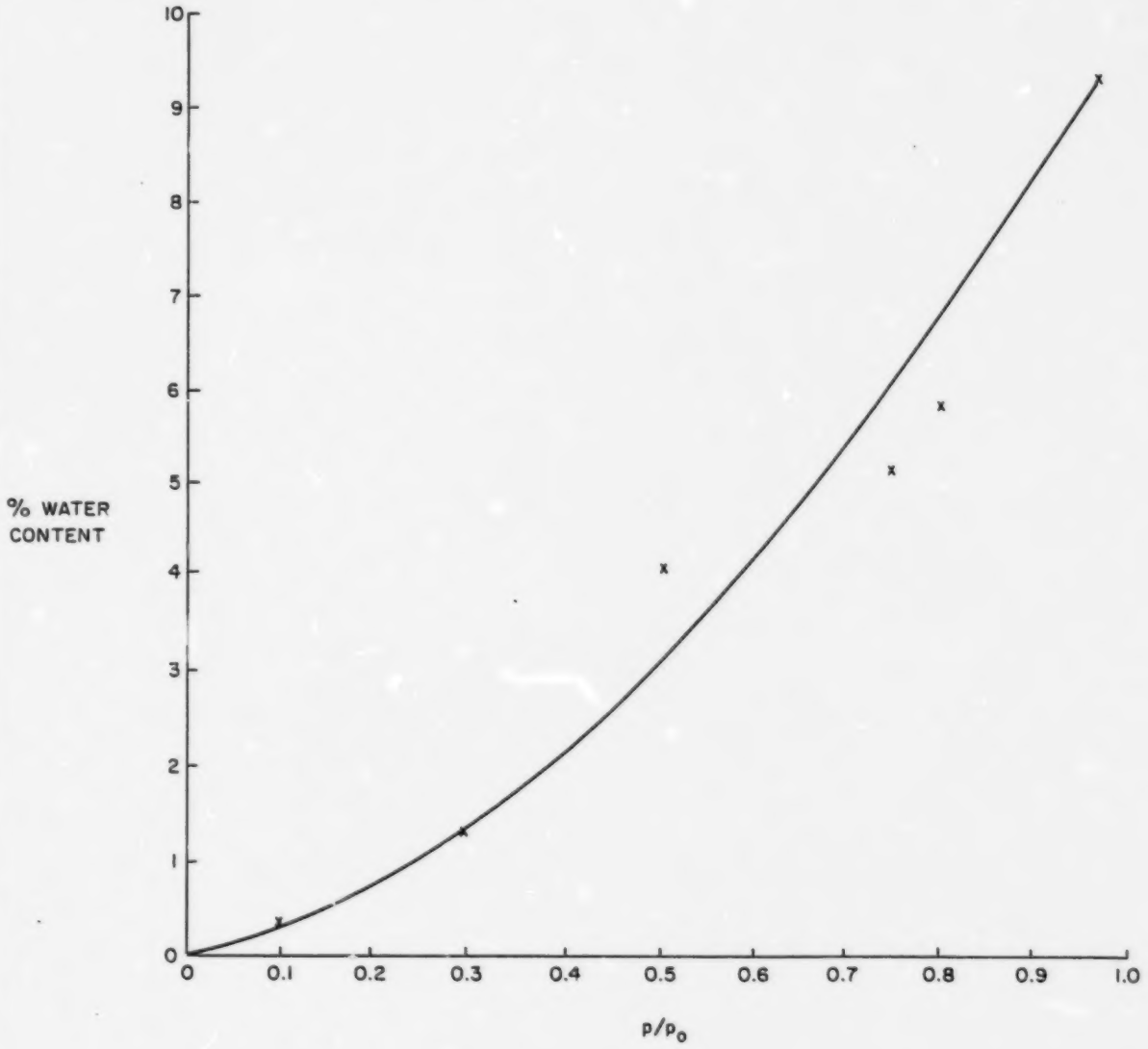




ADSORPTION ISOTHERM TEST GRAPHS FOR
LINGAN 6W-TOP AND LINGAN 9E-TOP



ADSORPTION ISOTHERM GRAPH FOR EVANS MINE



ADSORPTION ISOTHERM GRAPH FOR
SASKATCHEWAN BEARPAW SHALE

For the same relative humidity, the Evans seatearth adsorbed considerable more moisture than did the Lingan samples. The Bearpaw Shale adsorbed slightly more moisture than the Evans sample.

Ultrasonic Dispersion Tests

The Lingan samples failed to show any significant signs of dispersion even after twice the normal duration of the test. The behaviour of the Lingan samples is similar to that of a slate under the same conditions, i.e. no degradation over a reasonable length of time.

The Evans sample and the Bearpaw sample dispersed rapidly in the ultrasonic bath. However, the dispersion characteristics of the samples were quite different as a greater amount of -200 mesh fines were produced in the Bearpaw Shale. Figure 9 illustrates the dispersion characteristics of the Evans and the Bearpaw samples in normal tap water.

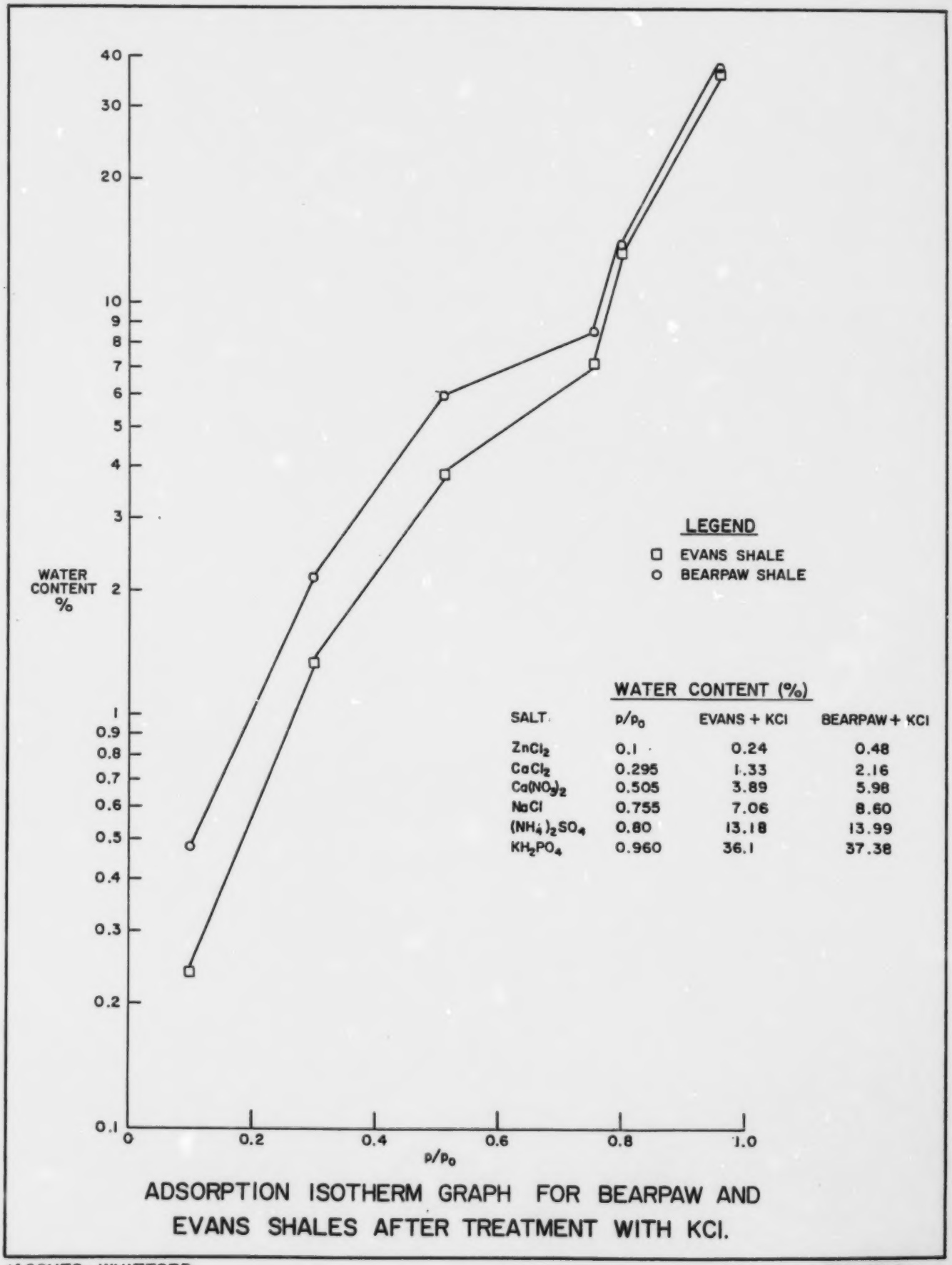
3.3 Aqueous Based Chemical Agents and Simulated Degradation of Problem Shales

This section presents the results of Adsorption Isotherm tests on Bearpaw and Evans Shales after treatment with potassium chloride, and a series of dispersion tests (as described in section 2.2) on problem shales, immersed in aqueous based solutions.

Results of Potassium Chloride Treated Samples

The adsorption isotherms for the Evans and Bearpaw samples treated with potassium chloride are shown in Figure 8a. The results of the tests indicate that over a wide range of relative humidities samples treated with potassium chloride adsorb more water than untreated samples.

The maximum amount of moisture adsorped by treated Evans shale in the 0.96 relative humidity desiccator after 30 days was in the order of three times as much as for



ADSORPTION ISOTHERM GRAPH FOR BEARPAW AND EVANS SHALES AFTER TREATMENT WITH KCl.

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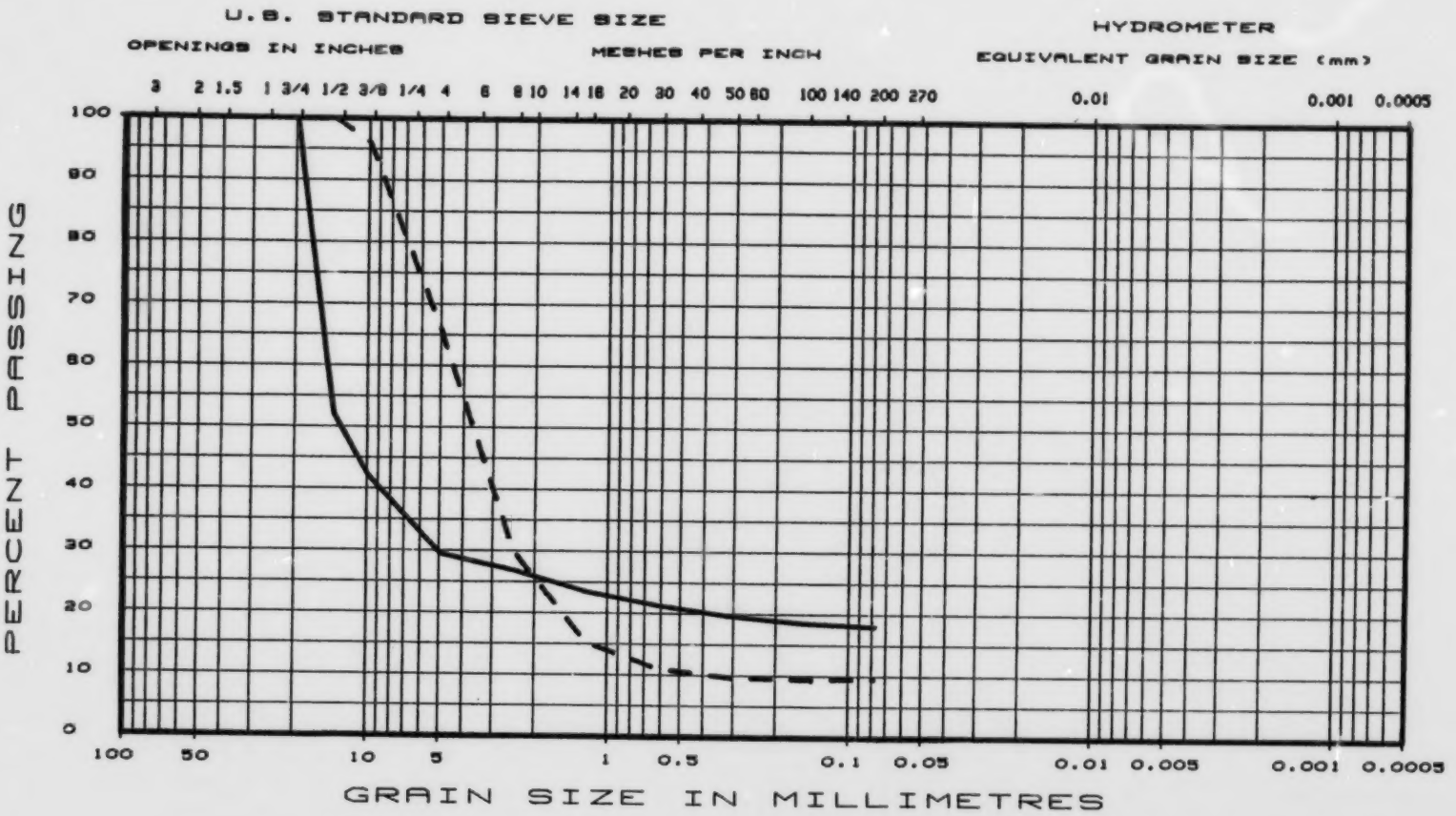
FIGURE 8a


U.S. STANDARD SIEVE SIZE
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EQUIVALENT GRAIN SIZE (mm)
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0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

Graph of Grain size Distribution of Evans and Bearpaw Shale in Normal Tap Water.

DESCRIPTION

—— BEARPAW SHALE IN NORMAL TAP WATER

— — EVANS SHALE IN NORMAL TAP WATER

untreated Evans. However, free water was observed to have accumulated showing adsorption well beyond the natural water content of the shale.

The rate of adsorption for the KCl-treated samples is illustrated in Figure 8b for relative humidities of 0.755 and 0.96. The curves show that water uptake continues for about 20 days in the case of untreated samples, but continues beyond 80 days for the treated samples.

Ultrasonic Dispersion Tests

Potassium Chloride and Sodium Chloride: The particle size distributions of Evans Shale samples after ultrasonic dispersion in normal tap water, 7.9 percent potassium chloride (KCl) and 7.9 percent sodium chloride (NaCl) are shown in Figure 10. The mean (D_{50}) particle sizes are 8.5 mm, 5.9 mm and 4.7 mm respectively. A reduction in particle degradation clearly occurred in both the NaCl and KCl solutions.

The particle size distributions of the Bearpaw Shale after ultrasonic dispersion in normal tap water, 7.9 percent KCl, and 7.9 percent NaCl are shown in Figure 11. The mean (D_{50}) particle sizes are 32 mm, 20 mm, and 13 mm respectively. A substantial reduction in fines occurred in both NaCl and KCl salt solutions.

Polymers and Potassium Chloride: Evans shale was also subjected to ultrasonic dispersion using the recommended dosage (1 kg. per 1 cubic metre of solution) of *SS-100 polymer and varying concentrations (3, 5.4, 7.9 percent by weight) of KCl in the fluid bath. The particle size distributions of Evans shale resulting from these tests are shown in Figure 12. The figure indicates similar fine, particle size distributions for the 3 percent KCl and 7.9 percent KCl trials, however the 5.4 percent KCl trial has a significantly larger particle size distribution, i.e. D_{50} size 8 mm (5.4 percent KCl) compared to 6 mm (2.0 percent KCl and 7.9 percent KCl).

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* Brand Name - basic Polyacrylamide

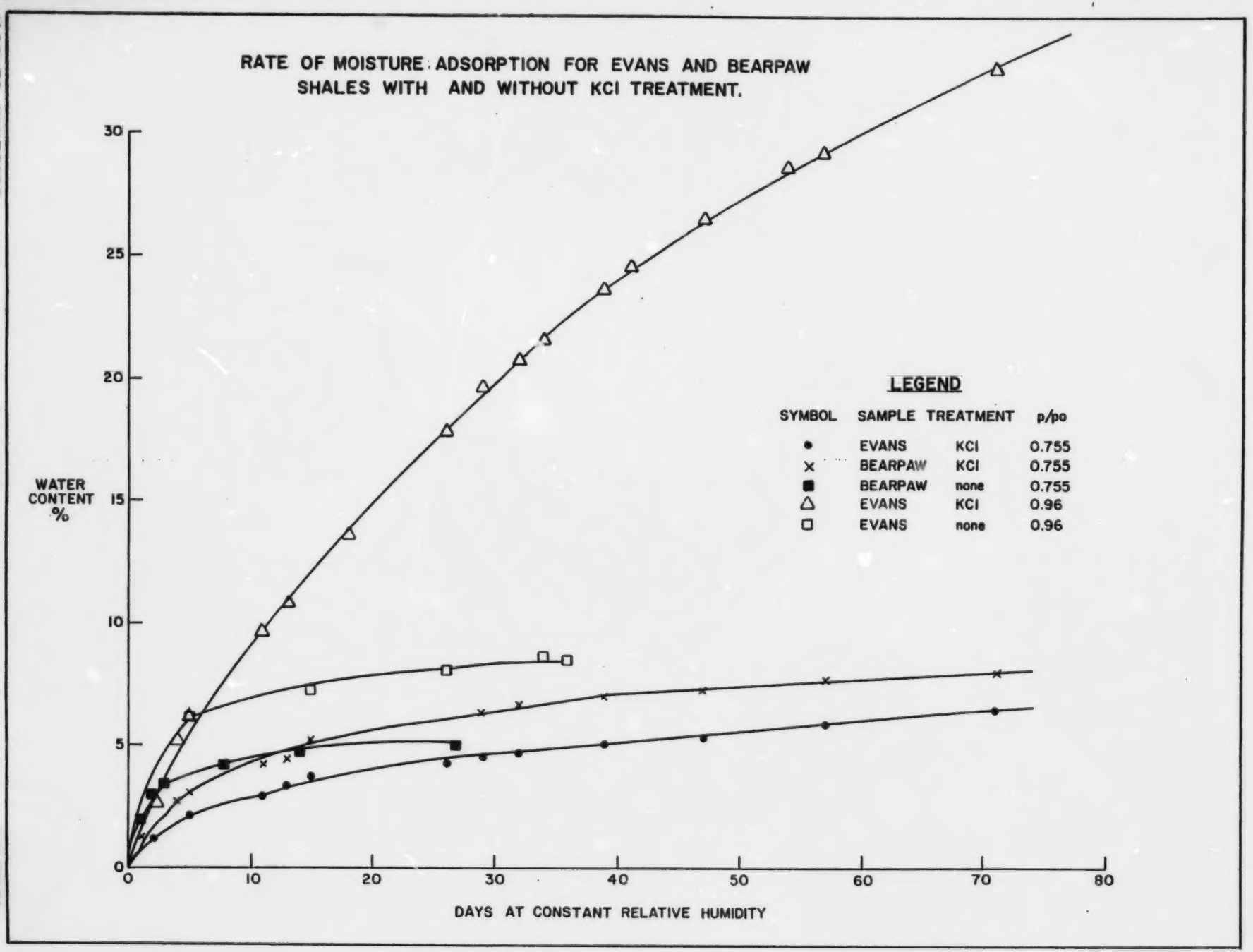
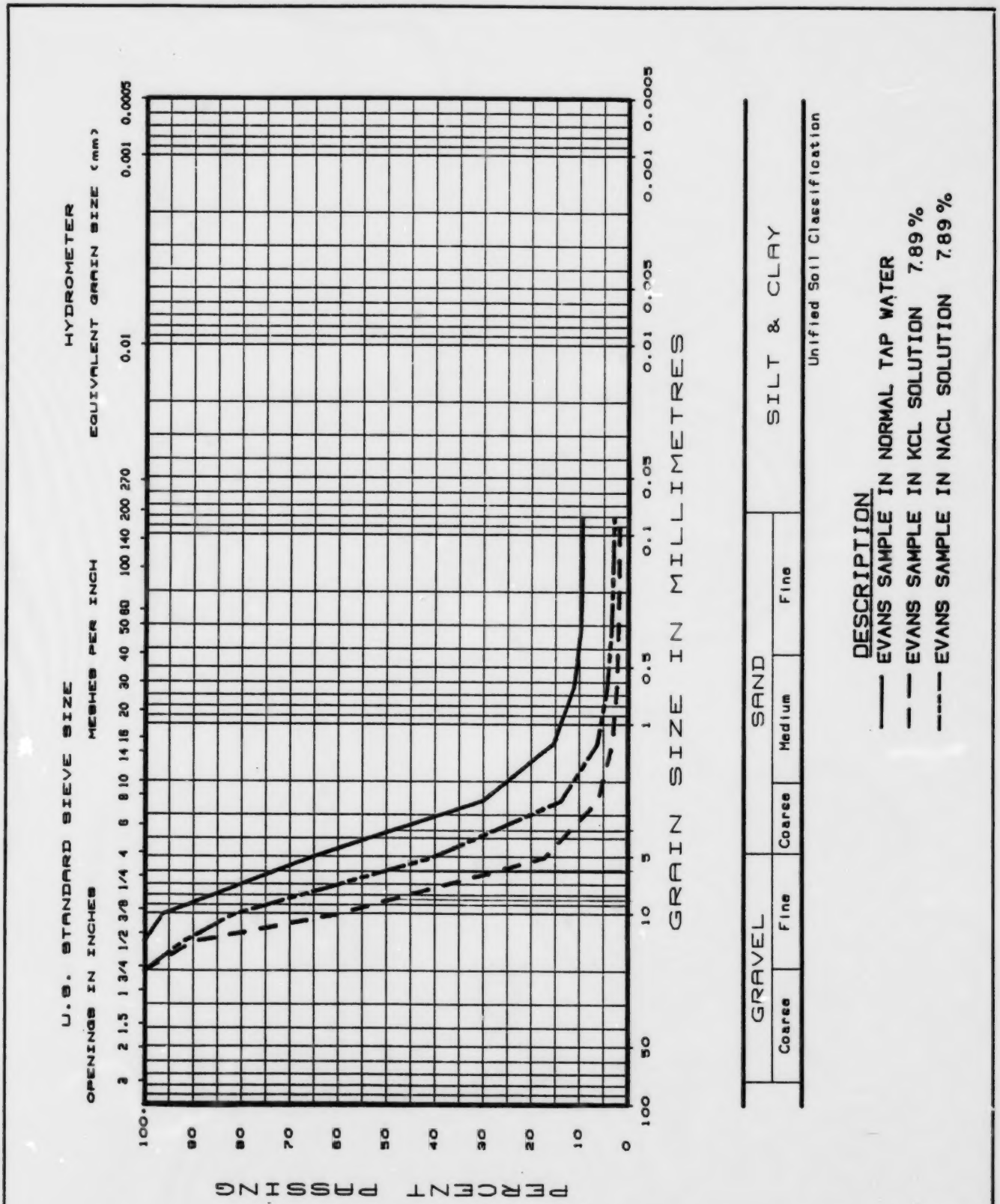
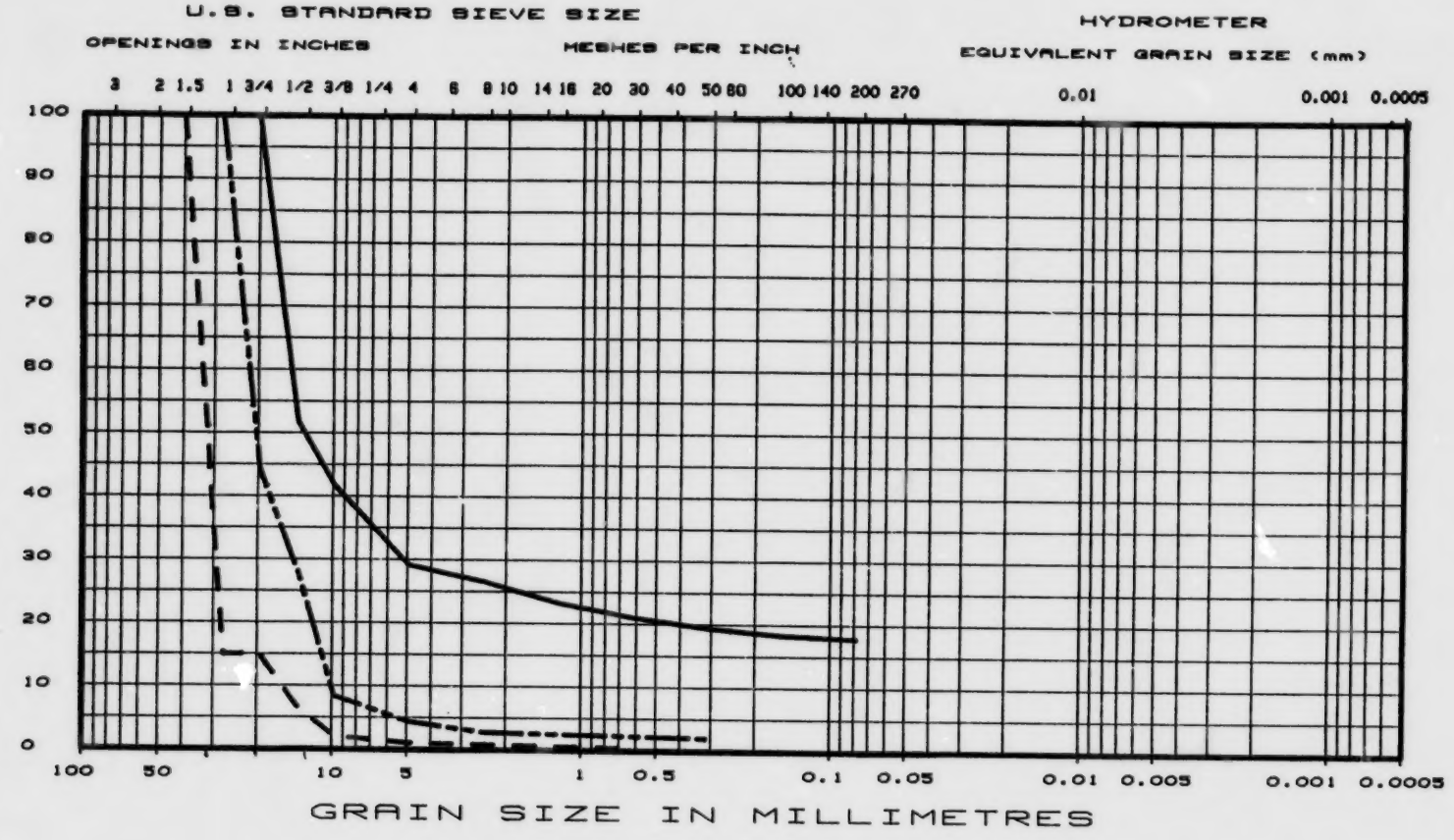

RATE OF MOISTURE ADSORPTION FOR EVANS AND BEARPAW SHALES WITH AND WITHOUT KCl TREATMENT.
WATER CONTENT %
0
5
10
15
20
25
30
0
10
20
30
40
50
60
70
80
DAYS AT CONSTANT RELATIVE HUMIDITY
LEGEND
SYMBOL SAMPLE TREATMENT p/po
EVANS KCl 0.755
BEARPAW KCl 0.755
BEARPAW none 0.755
EVANS KCl 0.96
EVANS none 0.96

FIGURE 8b



Graph of Grain Size Distribution of Evans Shale in Normal Tap Water, KCl Solution and NaCl Solution.


U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 6 8 10 14 16 20 30 40 50 60 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

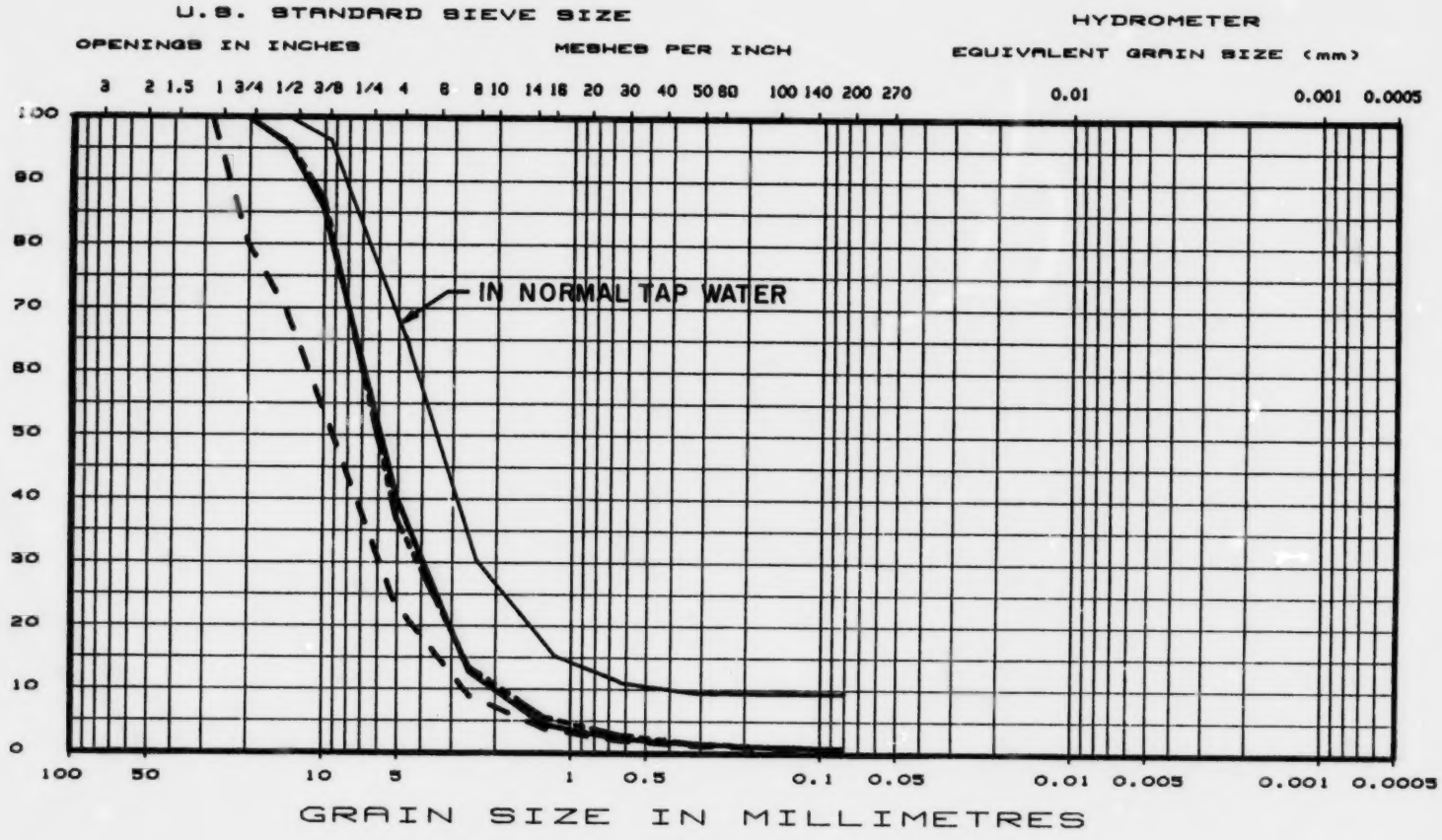
Unified Soil Classification

DESCRIPTION

—— BEARPAW SHALE IN NORMAL TAP WATER
– – BEARPAW SHALE IN KCL SOLUTION 7.89 %
·-·- BEARPAW SHALE IN NACL 7.89 %

Graph of Grain Size Distribution of the Bearpaw Shale in Normal Tap Water, KCl Solution and NaCl Solution.

JACQUES, WHITFORD 3-17 M1271 FIGURE 11


U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 6 8 10 14 16 20 30 40 50 80 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
IN NORMAL TAP WATER
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

Graph of Grain Size Distribution of Evans Shale in KCl and SS-100 Polymer Solutions.

DESCRIPTION

——— EVANS SHALE IN 3% KCL AND SS-100 POLYMER

– – – EVANS SHALE IN 5.4% KCL AND SS-100 POLYMER

—·—·— EVANS SHALE IN 7.89% KCL AND SS-100 POLYMER

JACQUES, WHITFORD 3-18 M1271 FIGURE 12

The Evans shale tests were repeated, substituting the recommended dosage of Alcomer* polymer for SS-100, and using the same concentrations as before (3, 5.4, and 7.9 percent by weight) of KCl. The particle size distributions of Evans shale resulting from these tests are shown in Figure 13. The figure indicates finer particle size distributions for the 3 percent KCl and 7.9 percent KCl trials than for the 5.4 percent KCl sample.

Figure 14 compares three trials with the Evans shale in 5.4 percent KCl and Alcomer, 5.4 percent KCl and SS-100, and 7.9 percent KCl solution. The plots show similar particle size distributions.

Figure 15 compares three trials with the Bearpaw Shale in 3 percent KCl and Alcomer, 3 percent KCl and SS-100, and 7.9 percent KCl solution. The plots show similar particle size distributions.

Calcium Chloride: The Evans shale was tested in a 4.75 percent $CaCl_2$ solution, the results are shown in Figure 16. The particle size distribution for the sample in the calcium chloride solution is finer than that for normal tap water.

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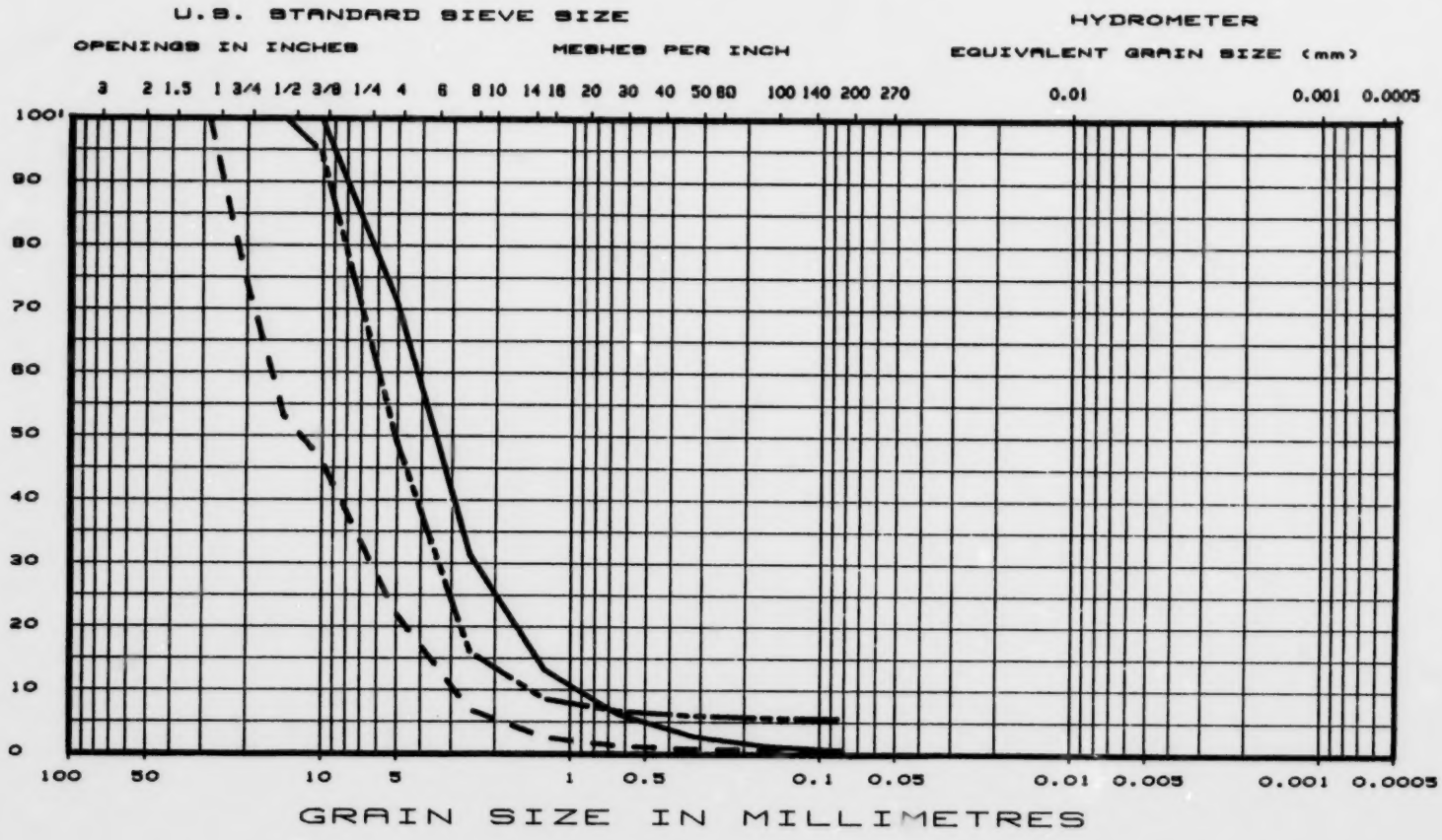
* Brand Name - basic Polyacrylamide


U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 6 8 10 14 16 20 30 40 50 60 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

DESCRIPTION

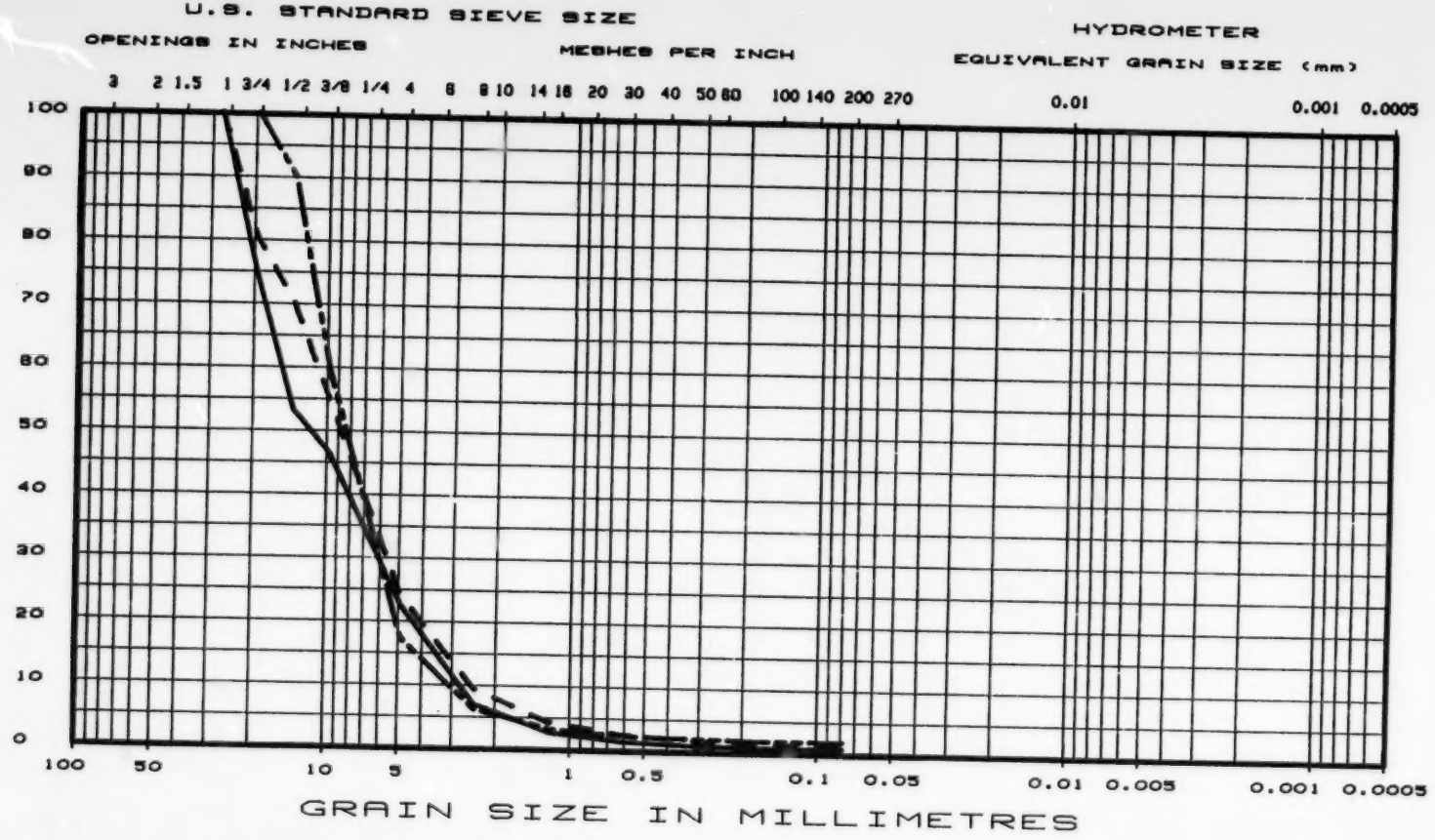
——— EVANS SHALE IN 3% KCL AND ALCOMER POLYMER

— — EVANS SHALE IN 5.4% KCL AND ALCOMER POLYMER

—·—·— EVANS SHALE IN 7.9% KCL AND ALCOMER POLYMER

Graph of Grain Size Distribution for Evans Sample in KCl and Alcomer Polymer Solutions.

JACQUES, WHITFORD 3-20 M 1271 FIGURE 13


U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 6 8 10 14 18 20 30 40 50 80 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

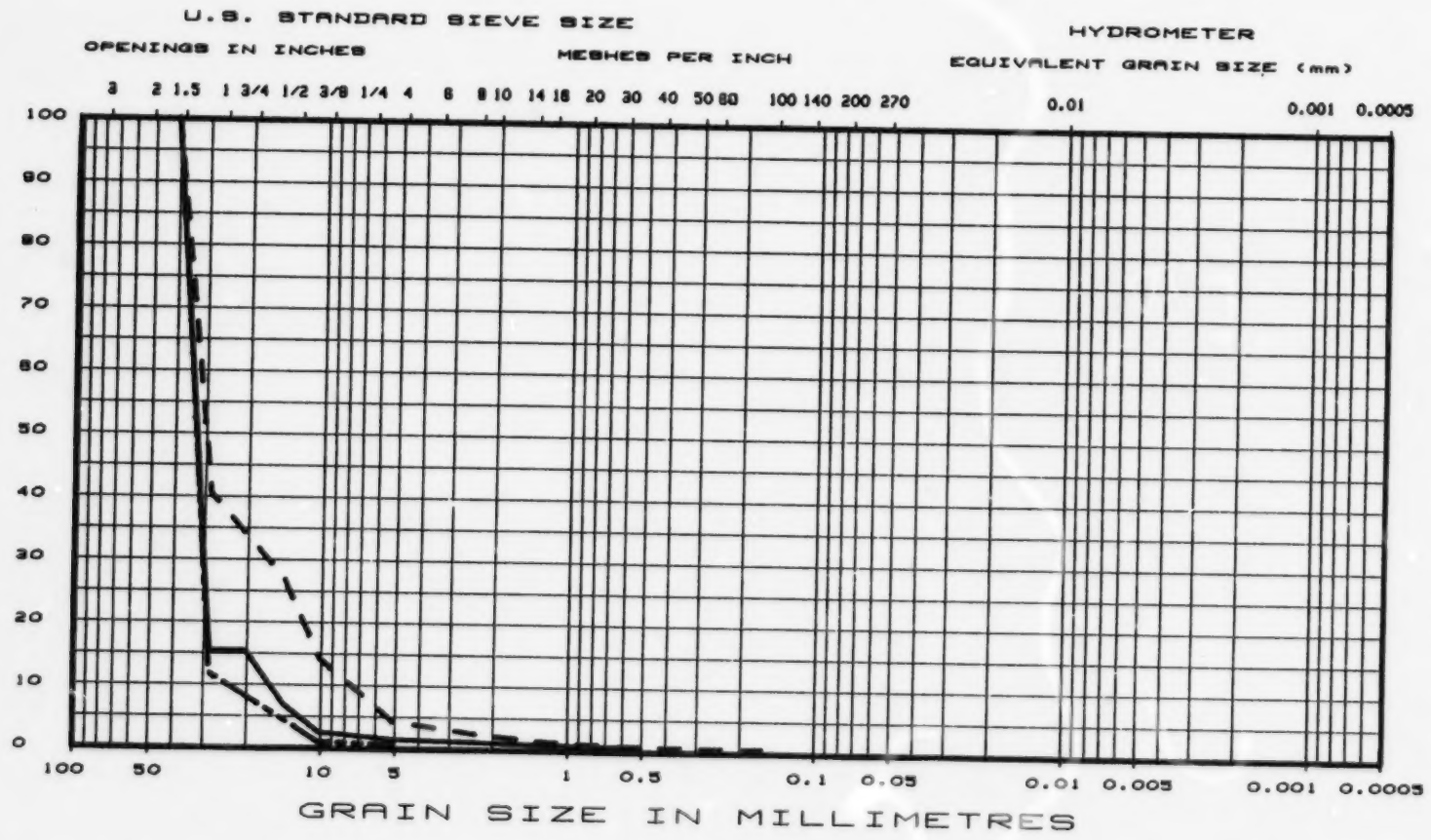
DESCRIPTION

——— EVANS SHALE IN 5.4% KCL AND ALCOMER

— — EVANS SHALE IN 5.4% KCL AND SS-100 POLYMER

— - — - EVANS SHALE IN 7.9% KCL SOLUTION

Graph of Grain Size Distribution of Evans Shale in 5.4% KCl Solution and Alcomer Polymer, 5.4% KCl and SS-100 Polymer and 7.9% KCl Solution.


U.S. STANDARD SIEVE SIZE
OPENINGS IN INCHES
MESHES PER INCH
HYDROMETER
EQUIVALENT GRAIN SIZE (mm)
3 2 1.5 1 3/4 1/2 3/8 1/4 4 6 8 10 14 18 20 30 40 50 60 100 140 200 270
0.01 0.001 0.0005
PERCENT PASSING
100 90 80 70 60 50 40 30 20 10 0
100 50 10 5 1 0.5 0.1 0.05 0.01 0.005 0.001 0.0005
GRAIN SIZE IN MILLIMETRES

GRAVEL		SAND			SILT & CLAY
Coarse	Fine	Coarse	Medium	Fine	

Unified Soil Classification

DESCRIPTION

——— BEARPAW SHALE IN 7.8% KCL SOLUTION

— — — BEARPAW SHALE IN 3% KCL AND ALCOMER POLYMER

—·—·— BEARPAW SHALE IN 3% KCL AND SS-100 POLYMER

Graph of Grain Size Distribution of Bearpaw Shale in 3% KCl and Alcomer Polymer, 3% KCl and SS-100 Polymer and 7.9 KCl Solution.

JACQUES, WHITFORD 3-22 M1271 FIGURE 15

Graph of Grain Size Distribution of the Evans Shale in 4.76% $CaCl_2$ Solution, Normal Tap Water, and 7.9% KCl Solution.

4.0 DISCUSSION OF RESULTS

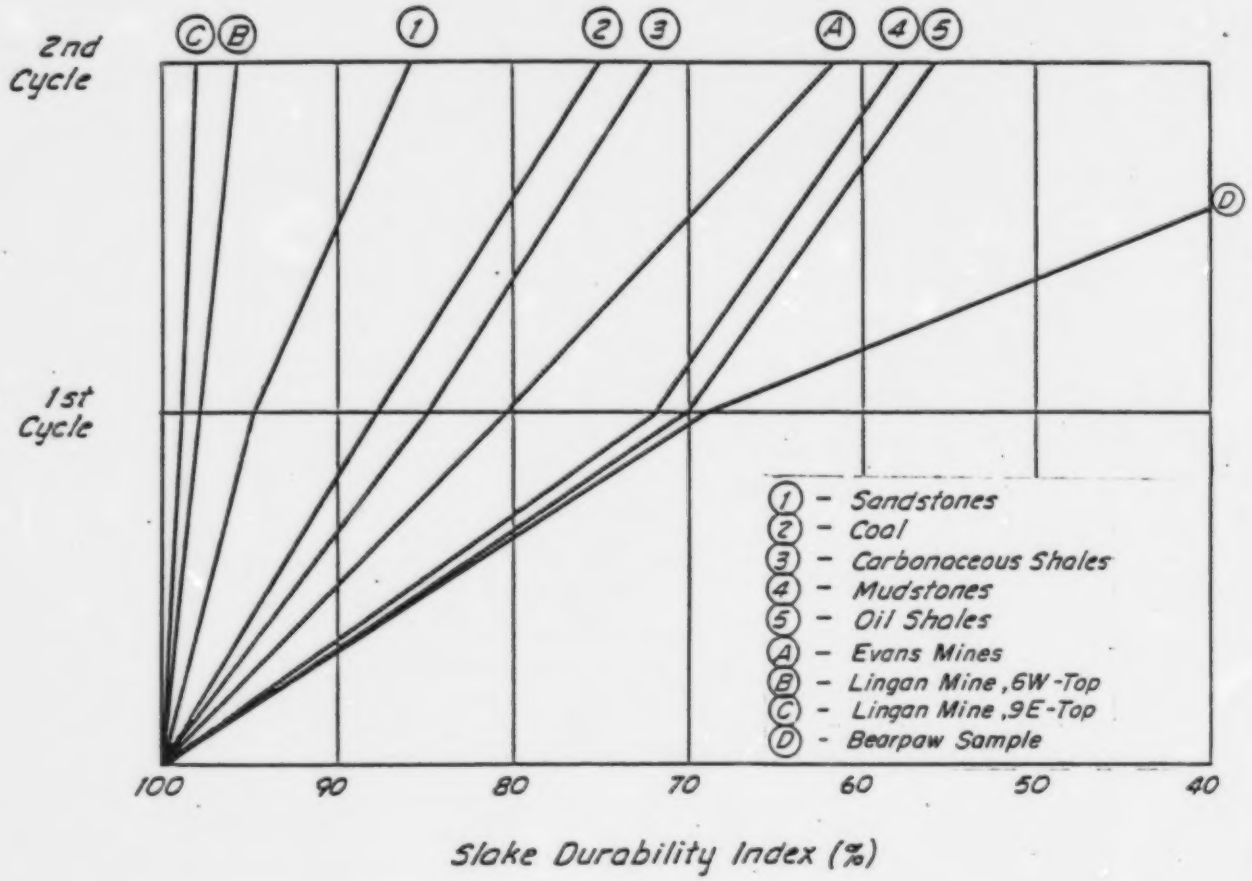
A visual comparison of the three seatearths from Cape Breton did not reveal distinct differences in appearance. The Evans shale shows significant traces of pyrite. In general, pyrite is prone to oxidation and is an attributable reason for swelling.

The Saskatchewan Bearpaw Shale is a much softer and less dense shale than the Evans and Lingan samples. This shale has a high moisture content (23.2% to 25.7%).

The Lingan and Evans samples generally show a low moisture content, extremely low porosity, a dry density in the 2.45 and 2.75 g/cm^3 bracket, and a low to non-existent plasticity. The Evans mine seatearths showed weakening and disintegration when subjected to cycles of wetting and drying in the slake durability test, and thus a low slake durability index (Figure 17). This low slake durability index of the Evans Shale may be attributed to its brittle, microfractured structure. The strong impermeable Lingan samples are highly resistant to slaking.

The Saskatchewan Bearpaw Shale of high moisture content, porosity, and plasticity index with a dry density of 2.71 gm/cm^3, is a well known swelling clay. The swelling properties are generally attributed to the high montmorillonite content of the clay fraction. The Bearpaw Shale showed rapid disintegration when subjected to cycles of wetting and drying in the slake-durability test. The slake durability index of the Bearpaw was much lower than the Evans shale. This reflects the weaker nature of the Bearpaw.

The disintegration of the Bearpaw was on the water shale boundary (surface) of the samples and not along fractures planes as in the Evans sample.


2nd Cycle
1st Cycle
C B 1 2 3 A 4 5 D
100 90 80 70 60 50 40
Slake Durability Index (%)
1 - Sandstones
2 - Coal
3 - Carbonaceous Shales
4 - Mudstones
5 - Oil Shales
A - Evans Mines
B - Lingan Mine, 6W-Top
C - Lingan Mine, 9E-Top
D - Bearpaw Sample

SLAKE DURABILITY TEST RESULTS AND
MAIN STRATA UNITS IN PICTOU COAL FIELD

Preliminary examination of Lingan and Evans samples when immersed in water showed no significant signs of swelling. However, extended periods of soaking of the Evans sample revealed dispersion and slight swelling. Even after extended soaking the Lingan samples showed no signs of swelling or dispersion. Subsequent laboratory testing confirmed the Lingan samples are non-problem seatearths.

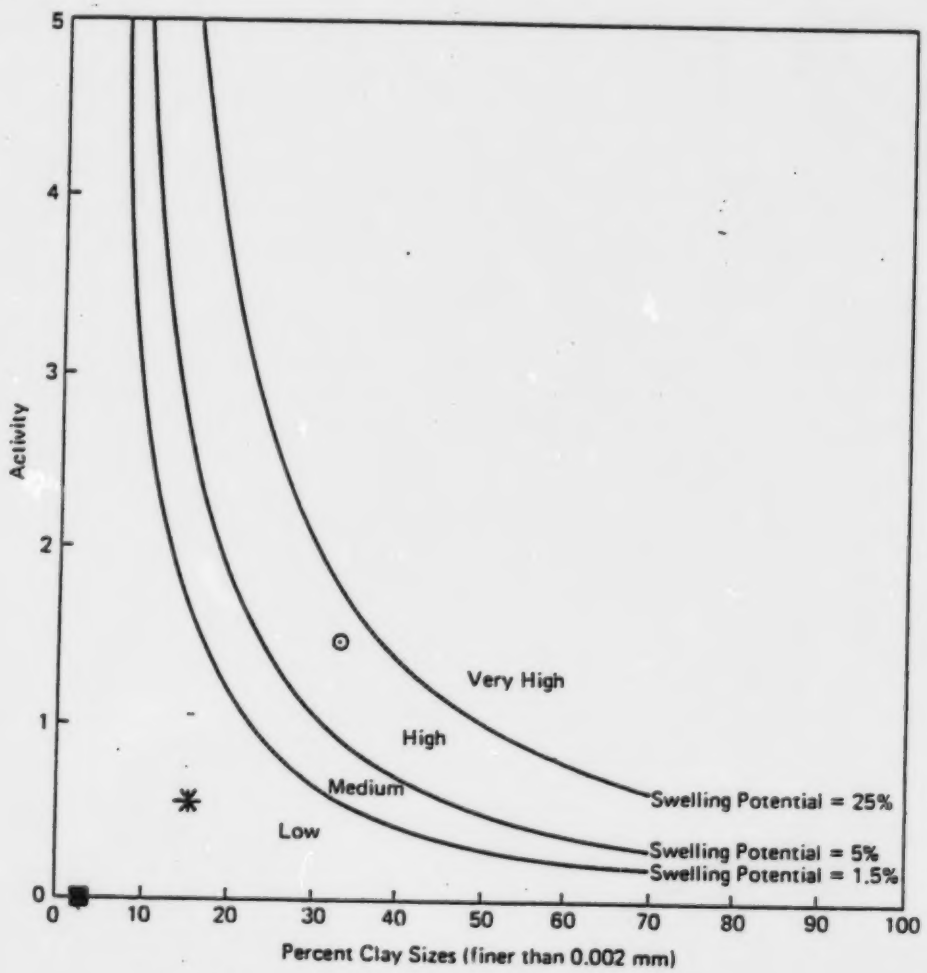
The Atterberg limits reflect the influence of both the type and amount of clay. To separate these influences the activity can be used (Skempton, 1953). Activity is defined as the ratio of plasticity index to clay size fraction (<2 microns), and is empirically related to clay mineral type.

Computed values of activity are shown below:

Sample	Plasticity Index	Clay Fractions	Activity
Lingan	0	3	0
Evans	81	15	0.55
Bearpaw	49	33	1.47

The activity of kaolinite is approximately 0.5. The clay mineral of the Evans sample may therefore be kaolinite. The high activity of the Bearpaw Shale may be attributed to montmorillonite clay. Seed, Woodward, and Lundren[2] (1962) suggest that for the activity and percent of clay sizes shown above, the Evans shale will have low swelling potential, the Bearpaw Shale will have a high swelling potential, and the Lingan will be non-swelling shale. This is illustrated in Figure 18.

The adsorptive isotherm test is a measure of the tendency of the shales to swell under varing ambient humidites. Entering the graphs (Figures 5 to 8) with the natural mositure content of the rocks, we can estimate the equilibrium relative humidity. At higher values, the shale will tend to swell and soften. For the Cape Breton shales, equilibrium relative



Classification chart for swelling potential (Seed, Woodward, and Lundgren, 1962).

* EVANS MINE

Activity 0.55
Clay Size 15%

⊙ BEARPAW SAMPLE

Activity 1.47
Clay Size 33%

■ LINGAN

Activity 0
Clay Size 3%

humidites are about 80 percent for the Lingan shales and 70 percent for the Evans sample, which indicates that these would exhibit limited swelling under ambient humidites in the mines. The test results for the Bearpaw Shale show much lower water contents than the initial values, and it is inferred that this material has undergone irreversible changes during over drying, thus, invalidating the test procedure.

Under laboratory conditions , the Evans sample absorbed 4 percent water by weight above its natural water content. The Lingan samples do not have a high potential for swelling as each gained less than 1 percent by weight above their natural water content. The Bearpaw Shale absorbed slightly more water than the Evans sample but much less than its natural water content.

The Bearpaw and Evans shales when treated with potassium chloride show a sharp increase in moisture adsorped. This increased moisture content may be attributed to a higher activity level of the shales created by the addition of potassium chloride.

Free water was observed to have accumulated in the Evans shale container in the 0.96 relative humidity desiccator and continued to accumulate. This accumulation of free water beyond the saturation of the shale suggests this test was invalidated as the characteristics of the shale could not be controlling adsorption.

The Evans sample exhibits disintegration upon exposure to water, thus potential problem behavior. The Saskatchewan Bearpaw Shale exhibits swelling and dispersion properties; those characteristics would make for a problem seatearth.

The mineralogy of the Lingan samples is believed to be quartz, illite, and kaolinite with some minor chlorite. The mineralogy of the Evans sample is similar to the Lingan samples, with major minerals consisting of quartz, illite, and kaolinite. There was no evidence of chlorite; however, pyrite is a minor mineral and has already been identified as a swelling source.

Montmorillonite, illite, kaolinite, and quartz appear to be the major minerals comprising the Bearpaw sample.

The mechanical degradation and disintegration of the Bearpaw Shale and Evans shale were reduced significantly when certain salts and polymers were added to the water. The relative effectiveness is shown in the following Table. Calcium chloride did not reduce degradation and may contribute to degradation.

Effectiveness of Chemical Treatment in Reducing Particle Degradation

Material	Effectiveness		
	Greatest	Mid-Range	Least
Evans shale	5.4% KCL & Alcomer	7.9% KCl & SS-100	
	7.9% KCl	7.9% NaCl	7.9% KCl & Alcomer
	5.4% KCl & SS-100	3% KCl & SS-100	3% KCL & Alcomer
Bearpaw Shale	7.9% KCl	3% KCl & Alcomer	7.9% NaCl
	3% KCl & SS-100		

The addition of water-soluble polymers to the potassium chloride solutions did not produce significant increase in resistance to degradation compared to KCl solution alone. The polyacrylamides tested in the laboratory situation produced a slippery slime when in contact with water. This sliming effect covers the shale surface creating a membrane which reduces particle and water movement. However, the success preventing mechanical degradation may be overwhelmed by the hazards of a slippery floor which will be created if applied underground.

5.0 CONCLUSIONS AND RECOMMENDATIONS

The Evans, Lingan and Bearpaw samples have been classified and profiled, such that their composition and properties have been derived. The Lingan samples show no problem behavior and may be considered a non-problem seatearth. The Evans sample has shown rapid mechanical disintegration in the ultrasonic dispersion test, which could be considered as problem behavior. The Bearpaw Shale, known to be an example of a swelling clay, has provided insight into the swelling behavior of shales.

The degradation of problem shales was significantly reduced by the addition of various aqueous based chemicals. Sodium chloride and potassium chloride effectively reduced dispersion of the Evans and Bearpaw Shales. Increasing concentrations of these salts in solution improved the stability of the problem shales. Potassium chloride in solution was the most effective of the salts for controlling dispersion of the shales.

Polyacrylamides effectively aid in stabilizing problem shales. The positive effects of stabilization may be outweighed by the potential hazards of its slippery nature. Therefore, polyacrylamides are not recommended for use as a problem shale stabilizer.

Potassium chloride has been found to the single most effective water based chemical agent capable of controlling problem shales. This material may be applied underground to mine areas in either liquid or powdered form. While the application of potassium chloride will reduce disintegration, it will not stop it entirely.

The application of potassium chloride to unsaturated seatearth materials may increase the potential for seatearths to adsorb water when the materials are in the unsaturated condition.

The application of potassium chloride to saturated seatearth materials will increase the stability of seatearth materials and may significantly reduce the development of problem areas in new mine workings.

The ultrasonic dispersion test developed as part of this study was judged to be very successful in identifying potential degradation, since it induced more breakdown in the Evans sample in a few minutes than was achieved by soaking for several months. The procedure is very simple and quick and can easily be performed on a routine basis. However, it needs to be calibrated to actual seatearth degradation in situ.

Proposed Mine Program

To examine the effectiveness of potassum chloride in stabilizing mine workings, the following scheme is proposed:

A. Identify problem areas of the mine.
B. Sample and classify problem areas.
C. Treat the shale.
D. Monitor test areas.

A. Using the ultrasonic dispersion test devised as outlined in this report, test shales suspected for problem behaviour. This should be conducted by an individual familiar with problem areas of the mine, as well as knowledge in geology and familiar with the dispersion test procedure.

B. Once problem areas have been identified they should be sampled and classified. To classify the problem seatearths, the following laboratory tests should be conducted:

- water content
- slake durability
- grain size

C. In each area in which a problem seatearth has been recognized, an area of the shale should be treated, and an area left untreated.

D. Following treatment of problem areas, a proper monitoring program should be followed. The monitoring should include:

- regular visual inspections
- instrumentation for - mine closure
  - stress field
- record wetting and drying cycles prior to and after treatment
- periodic dispersion tests.

If the stability of problem seatearth areas is maintained by the addition of potassium chloride, then KCL may prove to be a cost effective method for stabilization of Coal Measures Strata.

REFERENCES

1. Chenevert, Martin E., and Dennis E. O'Brien, Stabilization of Sensitive Shales Using Inhibited Potassium-Based Drilling Fluids, Society of Petroleum Engineers of AIME, Journal of Petroleum Technology, September, 1973.

2. Mitchell, James D. Fundamentals of Soil Behavior, John Wiley & Sons, Incorporated, Toronto 1976.

APPENDICES

A Statement of Work

B Background Laboratory Data

C Standard Test Procedures

APPENDIX A-1

STATEMENT OF WORK:

1. Obtain samples of seatearths from Cape Breton seams from CBCRL. A total of 24 samples of first size or larger, sealed to prevent moisture loss, 10 kgs minimum from each site. The sampling sites will be determined by consultation between the contractors, S.A. and CBDC.

2. Perform classification tests on samples as follows:

   - water content
   - porosity and density
   - Atterberg limits
   - dispersion, and
   - grain size distribution

3. On the basis of the above tests identify the problem and non-problem seatearths.

4. In consultation with the Scientific Authority obtain samples of six seatearths including problem and non-problem types. Samples to be in the form of cuboidal chunks of 150 mm minimum dimension, sealed to prevent moisture loss.

5. Perform tests as follows to determine the characteristics of the seatearths:

   - X-ray diffraction analysis of mineral content
   - differential thermal analysis of mineral content
   - cation exchange capacity
   - exchangeable sodium
   - permeability
   - slake durability
   - adsorptive isotherm
   - swelling strain index, and
   - compressive strength

6. In consultation with the Scientific Authority identify a test or tests that best indicate the degradation properties of the samples.

7. For a range of chemical concentrations, and with suitable pH buffering and controls determined in consultation with the Scientific Authority, treat samples of problem and non-problem seatearths using Potassium, Calcium and Polyacrylamide type chemicals.

8. Perform the test(s) identified in step six on the treated samples to determine the effectiveness of the chemical treatments.

9. Identify a chemical agent for use on a stabilising agent in underground coal mines using as a minimum basis for the decision:

   - the results of the test program
   - the dosage required and feasibility of pH buffering in an underground mine
   - cost of the treatment, and
   - deleterious effects of the selected chemical on machine/steel wear or corrosion, toxicity, and environmental conditions

10. Include the results of all of the above in the final report.

JACQUES, WHITFORD

POROSITY & DENSITY
(CANMET)

Sample EVANS MINE — Date Nov 27 - 28, 1985

Location HALIFAX — Tested by ACS

Borehole — — Depth — — Sample No. 1

A = Tare B = Wet C = Dry D = Coated E = Volume of water displaced

Specimen No.	Tare No.	A	B	C	D	E	W_w	W_s	W_b	V_b	w %	γ	γ_d
20	STA 1	48.57	146.33	143.76	144.58	40.29	2.57	95.19	97.76	39.37	2.70	2.48	2.42
21	STA 2	48.51	106.3	105.01	106.10	23.08	1.29	56.5	57.79	21.85	2.28	2.65	2.59
22	STA 4	47.46	149.61	146.98	148.38	41.96	2.63	99.52	102.15	40.39	2.64	2.53	2.46
23	STA 5	47.98	119.37	117.67	118.73	31.17	1.70	69.69	71.39	29.98	2.44	2.38	2.32
24	STA 6	48.72	114.21	112.78	113.62	26.25	1.43	64.06	65.49	25.31	2.23	2.59	2.53
25	STA 7	48.93	180.39	177.43	178.45	54.61	2.96	128.5	131.46	53.46	2.30	2.46	2.40
26	STA 8	48.76	160.88	157.91	159.18	46.44	2.97	108.93	112.12	45.01	2.73	2.49	2.42

F = Weight of flask : 65.65

G = Weight of flask + water : 165.28

H = Weight of flask + sample : 99.51

J = Weight of flask + water + sample : 185.91

G_w = H − F = 33.86 γ_p = Unit weight of Paraffin = .908

$G_v = V\left[1-(J-H)/(G-F)\right]$ = 13.28 $\gamma_g = G_w / G_v$ = 2.55

Sample water content, w : 2.47 %

bulk density, γ : 2.51 g/cm³

dry density, γ_d : 2.45 g/cm³

Sample grains density, γ_g : 2.55 g/cm³

porosity, n : 3.92 %

degree of saturation, S_r : 100.0 %

Remarks : SEE PIT SLOPE MANUAL, CANMET REPORT 77-25 FOR PROCEDURE.

M 1271 FIGURE 19

240 6/85

JACQUES, WHITFORD

POROSITY & DENSITY
(CANMET)

Sample LINGAN MINE, 6W - TOP — Date Nov 29, 1985

Location HALIFAX — Tested by ACS

Borehole — — Depth — — Sample No. 2

A = Tare B = Wet C = Dry D = Coated E = Volume of water displaced

Specimen No.	Tare No.	A	B	C	D	E	W_w	W_s	W_b	V_b	w%	γ	γ_d
10	STA 1	48.62	223.80	222.74	224.39	68.55	1.06	177.16	175.18	66.47	0.61	2.63	2.62
11	STA 2	48.56	119.24	118.53	119.79	27.56	0.71	69.97	70.62	26.14	1.01	2.70	2.67
12	STA 4	47.46	103.42	103.08	104.26	21.95	0.34	55.62	55.96	20.62	0.62	2.71	2.70
13	STA 5	48.00	207.43	205.62	207.54	61.57	1.81	157.62	159.43	59.41	1.11	2.68	2.65
14	STA 6	48.73	249.77	247.43	249.12	77.05	2.34	198.70	201.04	75.15	1.18	2.68	2.64
15	STA 8	48.78	149.76	149.17	150.64	39.76	0.59	100.39	100.98	38.11	0.59	2.65	2.63

F = Weight of flask : 65.65

G = Weight of flask + water : 165.28

H = Weight of flask + sample : 95.33

J = Weight of flask + water + sample : 183.78

G_w = H − F = 29.68 γ_p = Unit weight of Paraffin = 0.908

$G_v = V\left[1-(J-H)/(G-F)\right]$ = 11.22 $\gamma_g = G_w / G_v$ = 2.65

Sample water content, w : 0.85 %

bulk density, γ : 2.67 g/cm³

dry density, γ_d : 2.65 g/cm³

Sample grains density, γ_g : 2.65 g/cm³

porosity, n : 0.0 %

degree of saturation, S_r : 100.0 %

Remarks :

M 1271 FIGURE 20

JACQUES, WHITFORD

POROSITY & DENSITY
(CANMET)

Sample LINGAN MINE, 9E - TOP — Date Nov. 28, 1985

Location HALIFAX — Tested by ACS

Borehole — — Depth — — Sample No. 3

A = Tare B = Wet C = Dry D = Coated E = Volume of water displaced

Specimen No.	Tare No.	A	B	C	D	E	W_w	W_s	W_b	V_b	w %	γ	γ_d
1	STA 1	48.58	127.50	127.40	129.33	30.72	0.33	78.59	78.92	28.29	0.42	2.79	2.78
2	STA 2	48.51	146.79	146.38	148.93	38.45	0.41	97.87	98.28	35.59	0.42	2.78	2.75
3	STA 4	47.47	160.46	160.02	162.59	43.76	0.44	112.55	112.99	40.87	0.39	2.76	2.75
4	STA 5	48.00	107.20	106.94	108.70	23.76	0.26	58.94	59.20	21.78	0.44	2.72	2.71
5	STA 6	48.73	201.47	200.85	204.42	58.90	0.59	152.15	152.74	54.92	0.39	2.78	2.77

F = Weight of flask : 65.65

G = Weight of flask + water : 165.28

H = Weight of flask + sample : 95.85

J = Weight of flask + water + sample : 184.44

$G_w = H - F =$ 30.2 γ_p = Unit weight of Paraffin = 2.75

$G_v = V\left[1-(J-H)/(G-F)\right] =$ 11.08 $\gamma_g = G_w / G_v =$ 2.75

Sample water content, w : 0.41 %

bulk density, γ : 2.77 g/cm³

dry density, γ_d : 2.75 g/cm³

Sample grains density, γ_g : 2.75 g/cm³

porosity, n : 0.0 %

degree of saturation, S_r : 100.0 %

Remarks :

M 1271 FIGURE 21

JACQUES, WHITFORD

POROSITY & DENSITY
(CANMET)

Sample SASKETCHEWAN BEARPAW SHALE Date MAY 12/86.

Location HALIFAX Tested by B.W.W

Borehole — Depth — Sample No. 4

A = Tare B = Wet C = Dry D = Coated E = Volume of water displaced

Specimen No.	Tare No.	A	B	C	D	E	W_w	W_s	W_b	V_b	w %	γ	γ_d
1	Z6	34.62	109.83	97.29	98.08	34	12.54	62.67	75.21	33.1	20.0	2.27	1.89
2	Z7	35.10	139.44	121.95	122.91	48.5	17.49	86.85	104.34	47.4	20.1	2.20	1.83
3	Z2	34.99	135.19	118.00	119.07	46	17.19	83.01	100.2	44.8	20.7	2.24	1.85
4	Z4	34.98	146.23	127.82	129.15	52	18.41	92.84	111.25	50.5	19.8	2.20	1.84
5	R1	34.80	114.66	100.92	101.72	37	13.74	66.12	79.86	36.1	20.7	2.21	1.83
6	STA 7	35.70	117.84	104.22	105.31	38.5	13.62	68.52	82.14	37.3	19.8	2.20	1.84

F = Weight of flask : 88.09

G = Weight of flask + water : 287.4

H = Weight of flask + sample : 138.21

J = Weight of flask + water + sample : 319.1

$G_w = H - F =$ 50.12 γ_p = Unit weight of Paraffin = .908

$G_v = V\left[1-(J-H)/(G-F)\right] =$ 18.48 $\gamma_g = G_w / G_v =$ 2.71

Sample water content, w : 20.2 %

bulk density, γ : 2.22 g/cm³

dry density, γ_d : 1.85 g/cm³

Sample grains density, γ_g : 2.71 g/cm³

porosity, n : 31.7 %

degree of saturation, S_r : 117.8 %

Remarks :

M 1271 FIGURE 22

EVANS MINE

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
.0638	0.0	.5	21.7	30.5	38.69
.0456	0.0	1.0	21.7	32.2	36.90
.0326	0.0	2.0	21.7	30.7	34.84
.0210	0.0	5.0	21.7	28.0	31.10
.0125	0.0	15.0	21.7	25.0	28.08
.0088	0.0	30.0	21.7	24.7	26.59
.0061	0.0	60.0	22.0	22.2	23.00
.0045	0.0	120.0	22.2	20.7	21.39
.0032	0.0	255.0	22.7	18.0	17.96
.0026	0.0	364.0	23.0	16.8	16.49
.0014	0.0	1408.0	22.0	14.7	13.03
.0007	0.0	5971.0	21.7	10.5	7.08
.0007	0.0	5971.0	21.7	10.5	7.08

Sample weight for hydrometer analysis = 69.50 grams.
Specific gravity used in hydrometer analysis = 2.65

BOREHOLE
LINGAN MINE

SAMPLE
6W - TOP

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
.0674	0.0	.5	21.0	22.5	19.63
.0481	0.0	1.0	21.0	21.2	18.14
.0481	0.0	1.0	21.0	21.2	18.14
.0343	0.0	2.0	21.0	19.7	16.59
.0219	0.0	5.0	21.0	18.4	14.87
.0200	0.0	6.0	21.0	18.0	14.41
.0130	0.0	15.0	21.0	14.2	9.98
.0092	0.0	30.0	21.5	12.0	8.82
.0065	0.0	64.0	21.7	11.0	6.59
.0044	0.0	135.0	22.0	9.0	4.41
.0033	0.0	245.0	22.5	8.0	3.49
.0025	0.0	410.0	23.0	7.0	2.56
.0014	0.0	1392.0	20.0	7.2	1.33
.0007	0.0	6139.0	21.7	6.2	1.00

Sample weight for hydrometer analysis = 66.40 grams.
Specific gravity used in hydrometer analysis = 2.65

BOREHOLE
LINGAN MINE

SAMPLE
9E - TOP

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
.0596	0.0	.5	21.0	33.5	33.29
.0430	0.0	1.0	21.0	32.0	31.16
.0430	0.0	1.0	21.0	32.0	31.16
.0324	0.0	2.0	21.0	24.0	21.70
.0211	0.0	5.0	21.0	19.7	16.62
.0125	0.0	15.0	21.0	15.5	11.63
.0089	0.0	30.0	21.5	13.2	9.18
.0064	0.0	60.0	21.7	11.2	6.92
.0064	0.0	60.0	21.7	11.2	6.92
.0045	0.0	120.0	22.2	9.5	5.16
.0031	0.0	228.0	22.7	9.2	5.05
.0025	0.0	390.0	23.0	7.5	3.19
.0014	0.0	1378.0	20.0	7.5	1.70
.0006	0.0	6120.0	21.7	6.2	1.01

Sample weight for hydrometer analysis = 70.10 grams.
Specific gravity used in hydrometer analysis = 2.70

Grain Size Data of Evans, Lingan 6W-TOP, Lingan 9E-TOP.

SLAKE - DURABILITY INDEX

Sample Evans Mine Date Nov 22-27 /1985

Tested by ACS

Location Halifax

Borehole - Depth -

Sample No. 1

A = drum + dry sample before test

B = drum + dry sample after first cycle

C = drum + dry sample after second cycle

D = drum

Weight A = 1200.0 g Weight C = 992.4 g

Weight B = 1094.4 g Weight D = 657.9 g

Slake - durability index, $I_{d2} = \frac{C-D}{A-D} \times 100\ \% = .617 \times 100 = 61.7\ \%$

Remarks :

SLAKE - DURABILITY INDEX

Sample LINGAN MINES 6W - TOP Date Nov 22-27/1985

Tested by ACS

Location HALIFAX

Borehole — Depth —

Sample No. 2

A = drum + dry sample before test

B = drum + dry sample after first cycle

C = drum + dry sample after second cycle

D = drum

Weight A = 1153.0 g Weight C = 1130.9 g

Weight B = 1143.3 g Weight D = 665.1 g

Slake - durability index, $I_{d2} = \frac{C-D}{A-D} \times 100\ \% =$ .954 100 = 95.4 %

Remarks :

JACQUES, WHITFORD M 1271 FIGURE 25

SLAKE - DURABILITY INDEX

Sample LINGAN MINES 9E: TOP Date Nov 22-27, 1985

Tested by ACS

Location HALIFAX, N.S.

Borehole - Depth -

Sample No. 3

A = drum + dry sample before test

B = drum + dry sample after first cycle

C = drum + dry sample after second cycle

D = drum

Weight A = 1155.0 g Weight C = 1145.4 g

Weight B = 1150.6 g Weight D = 656.6 g

Slake - durability index, $I_{d2} = \frac{C-D}{A-D} \times 100\ \% = \frac{.981}{} \times 100 = 98.1\ \%$

Remarks :

JACQUES, WHITFORD

M 1271 FIGURE 26

SLAKE - DURABILITY INDEX

Sample SASKETCHEWAN BEARPAW SHALE Date MARCH 21 / 1986

Tested by B.W

Location HALIFAX, N.S.

Borehole - Depth -

Sample No. 4

A = drum + dry sample before test

B = drum + dry sample after first cycle

C = drum + dry sample after second cycle

D = drum

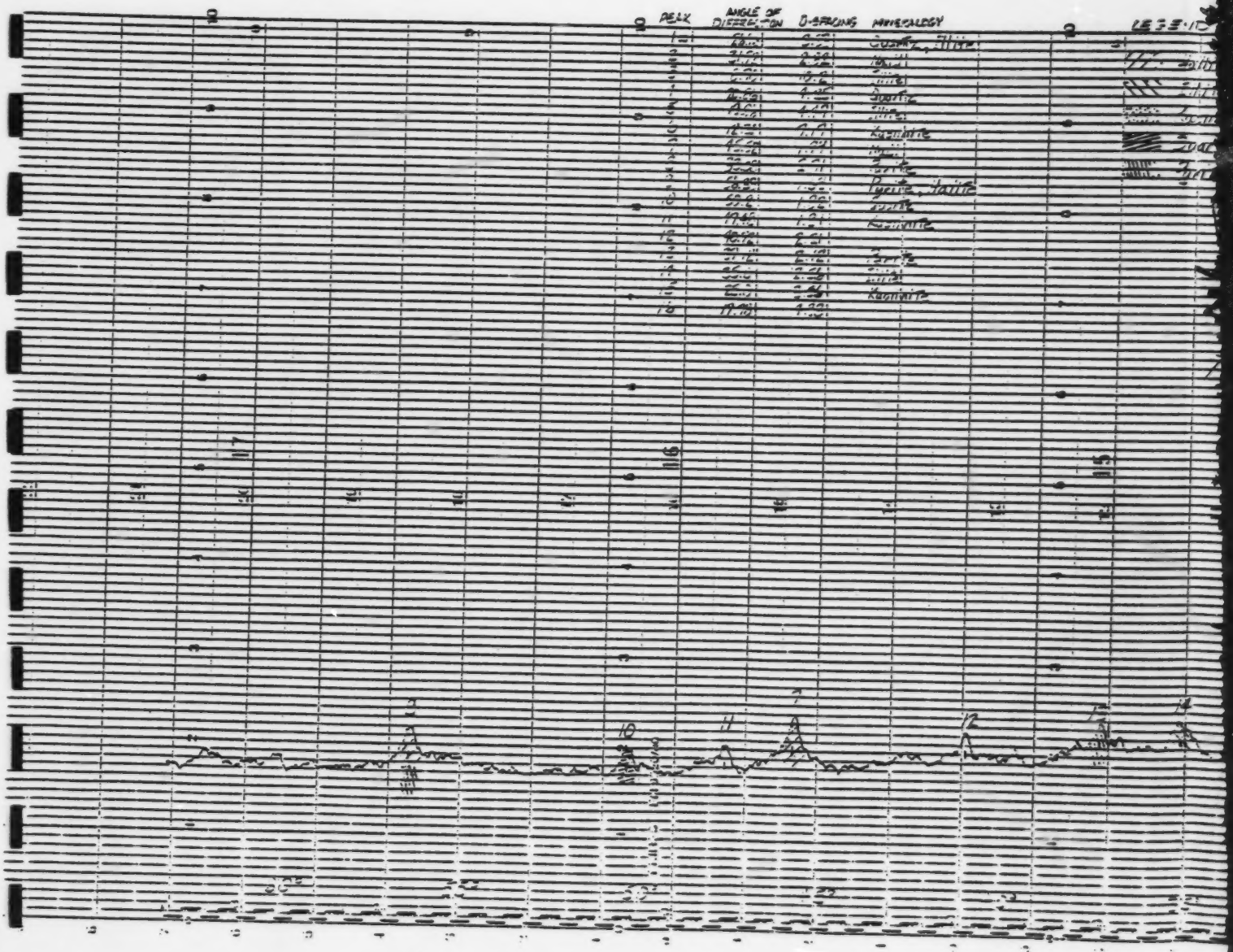
Weight A = 1063.2 g Weight C = 735.4 g

Weight B = 936.6 g Weight D = 656.1 g

Slake - durability index, $I_{d2} = \frac{C-D}{A-D} \times 100\ \% =$ .195 100 = 19.5 %

Remarks :

JACQUES, WHITFORD M 1271 FIGURE 27


PEAK
ANGLE OF DIFFRACTION
D-SPACING
MINERALOGY

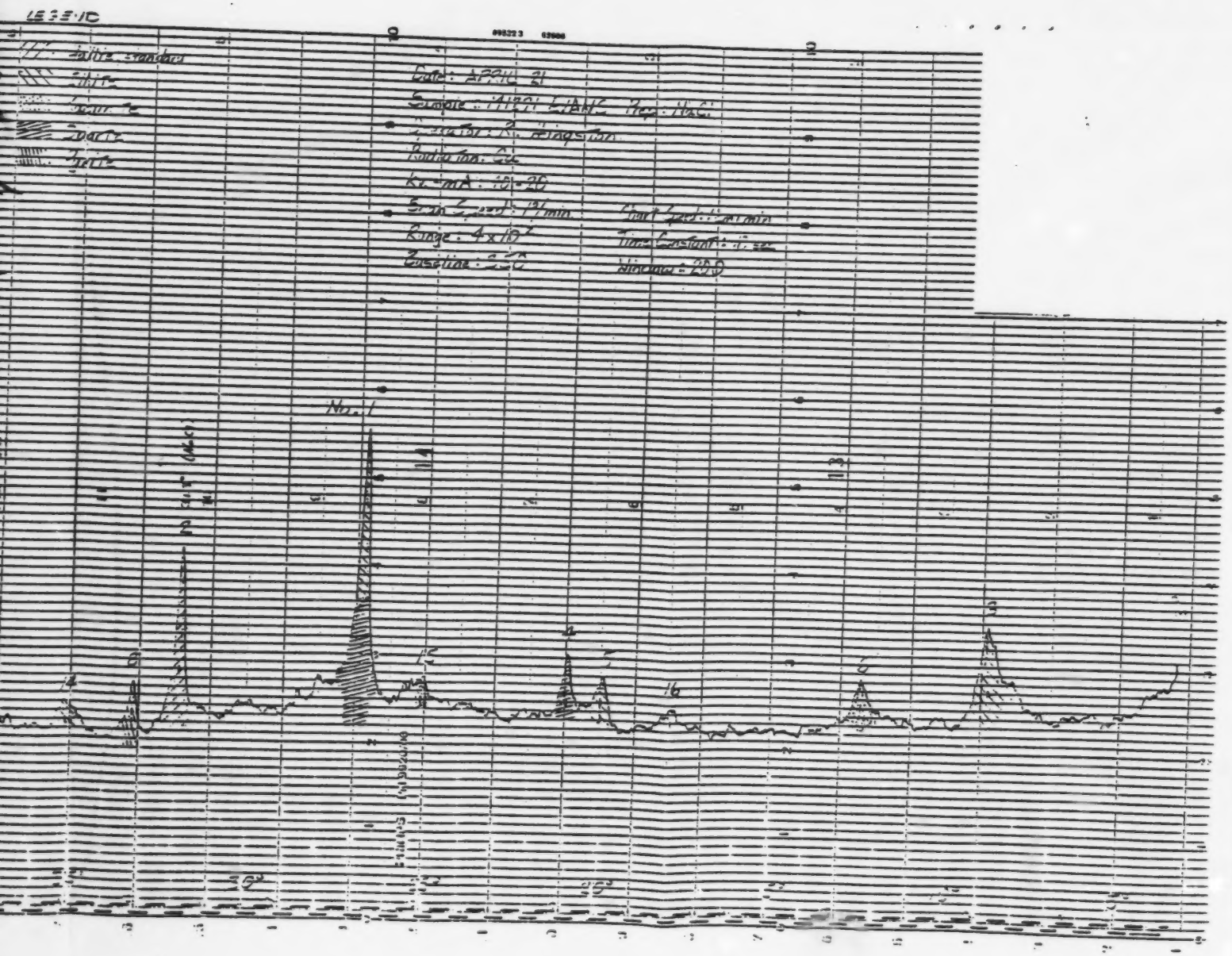
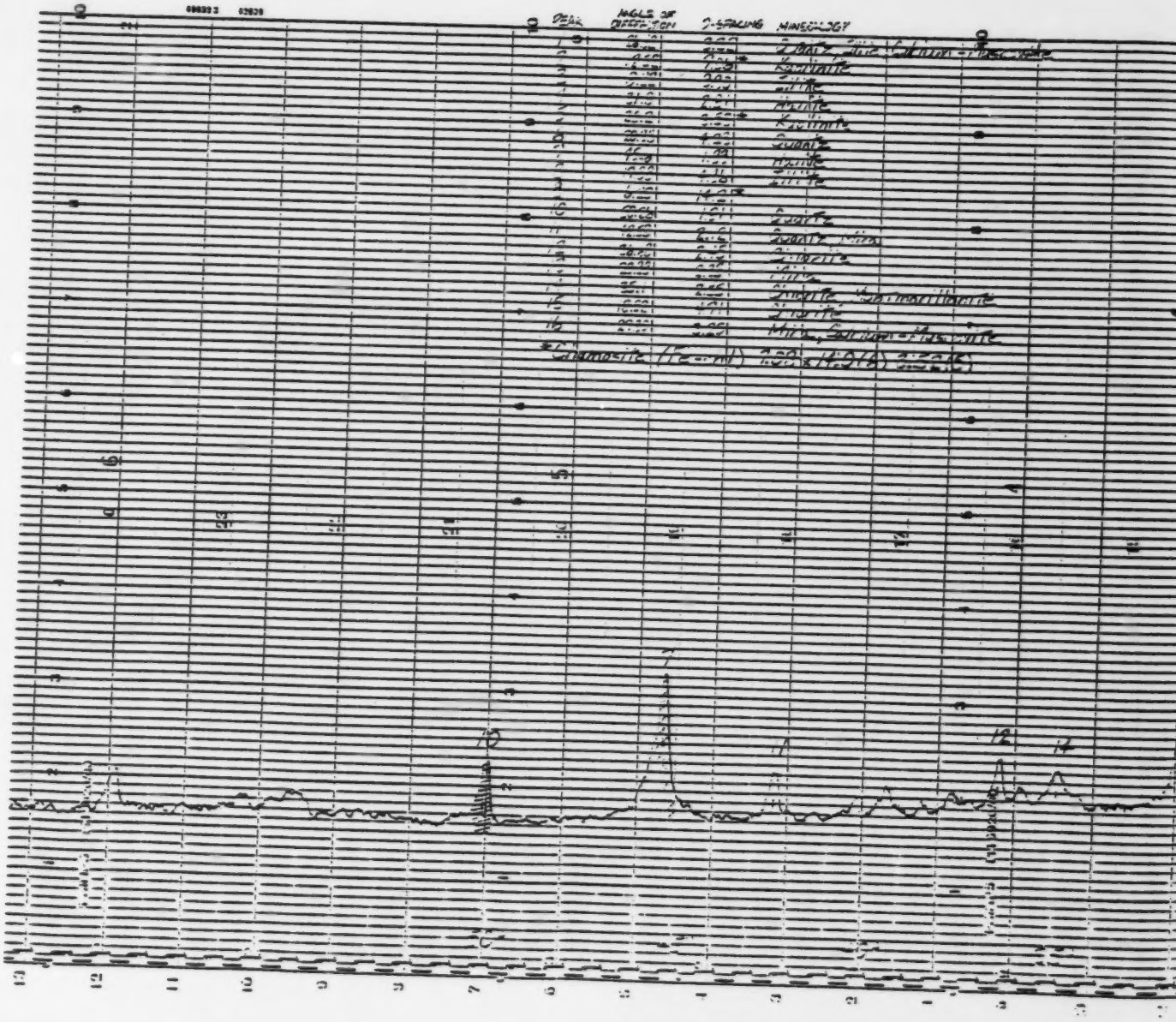


FIGURE 28: X-RAY DIFFRACTOGRAMS OF EVANS SHALE


PEAK
ANGLE 2θ DEFLECTION
D-SPACING
MINERALOGY

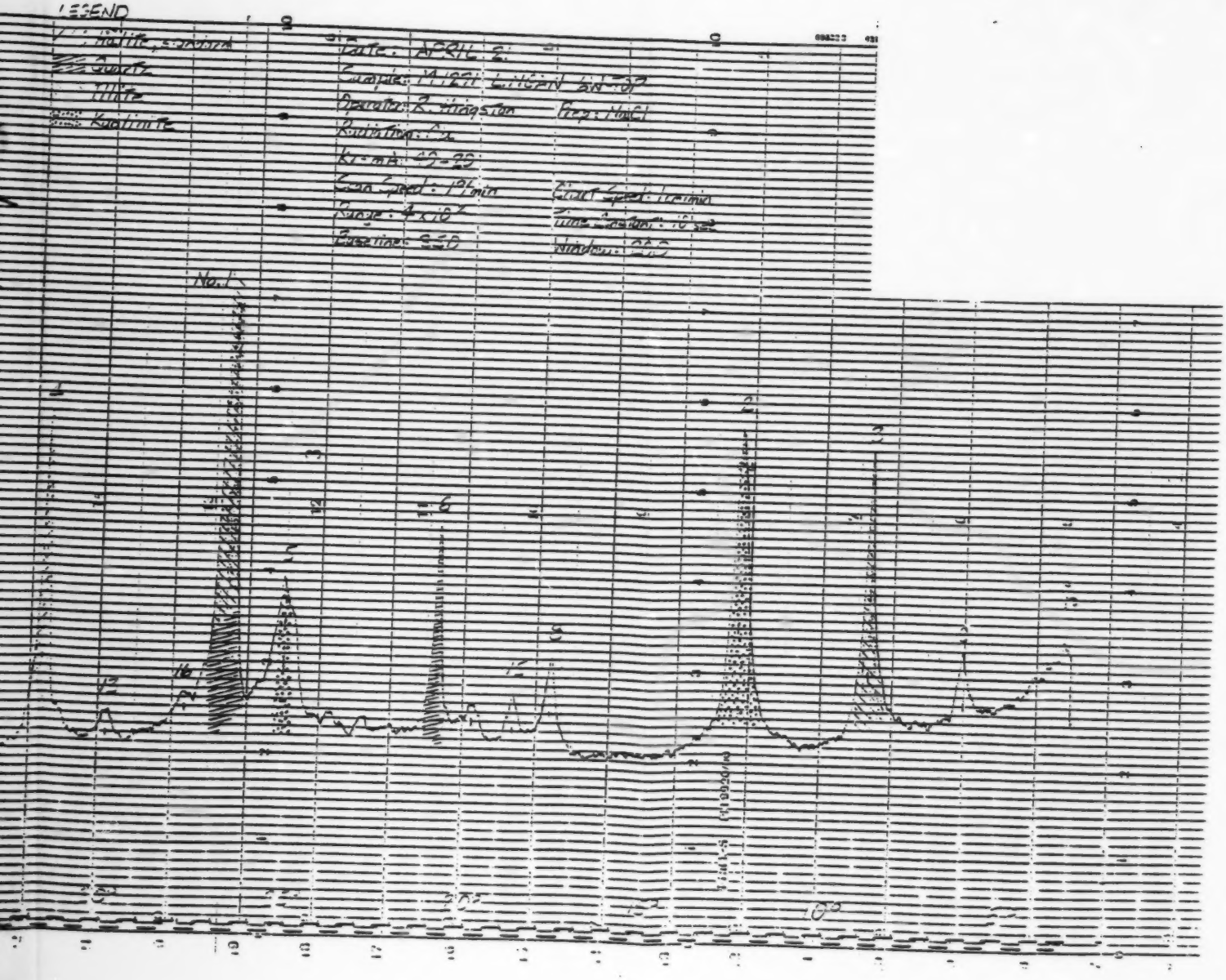
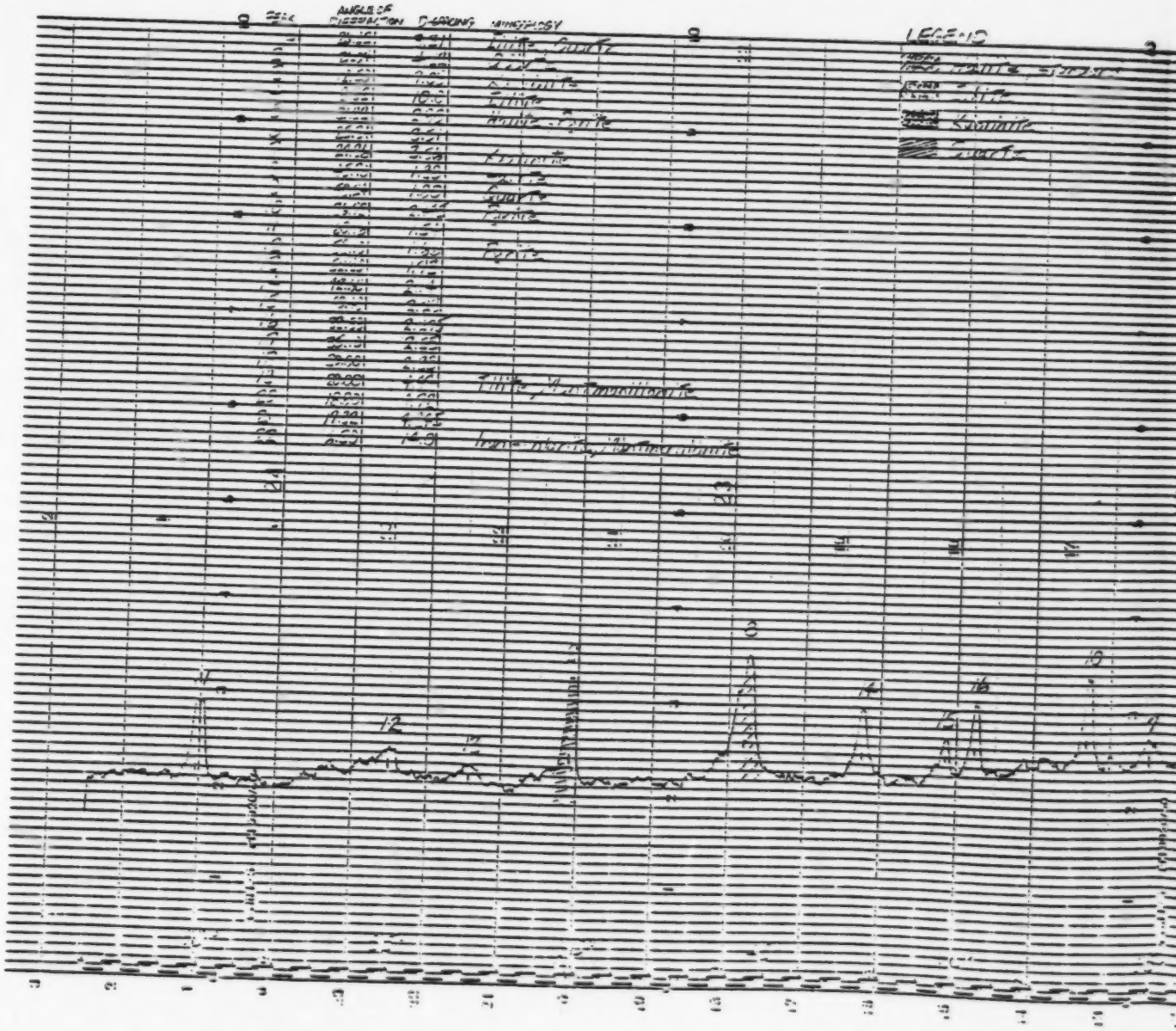


FIGURE 29 : X-RAY DIFFRACTOGRAMS OF LINGAN 6W-TOP SHALE



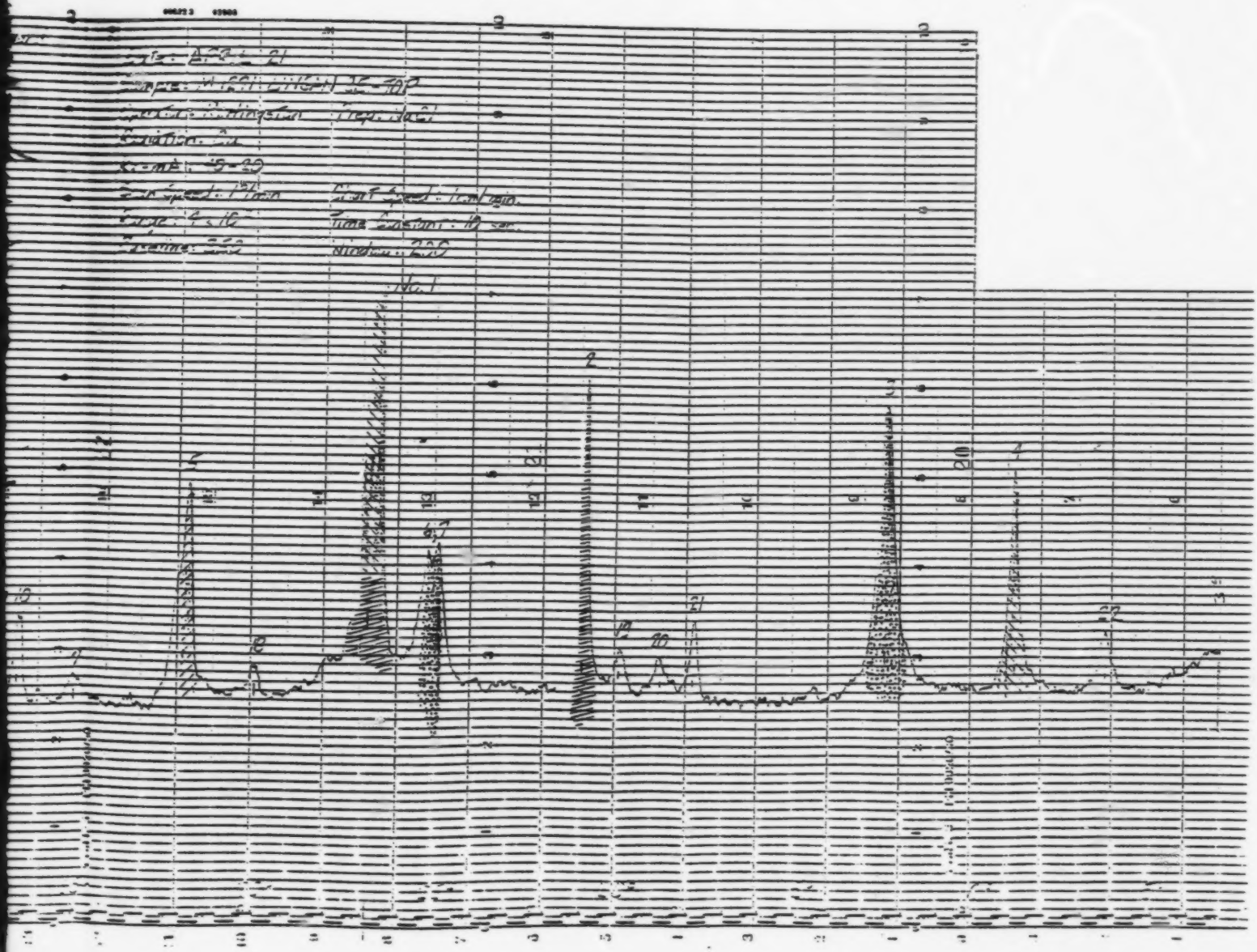
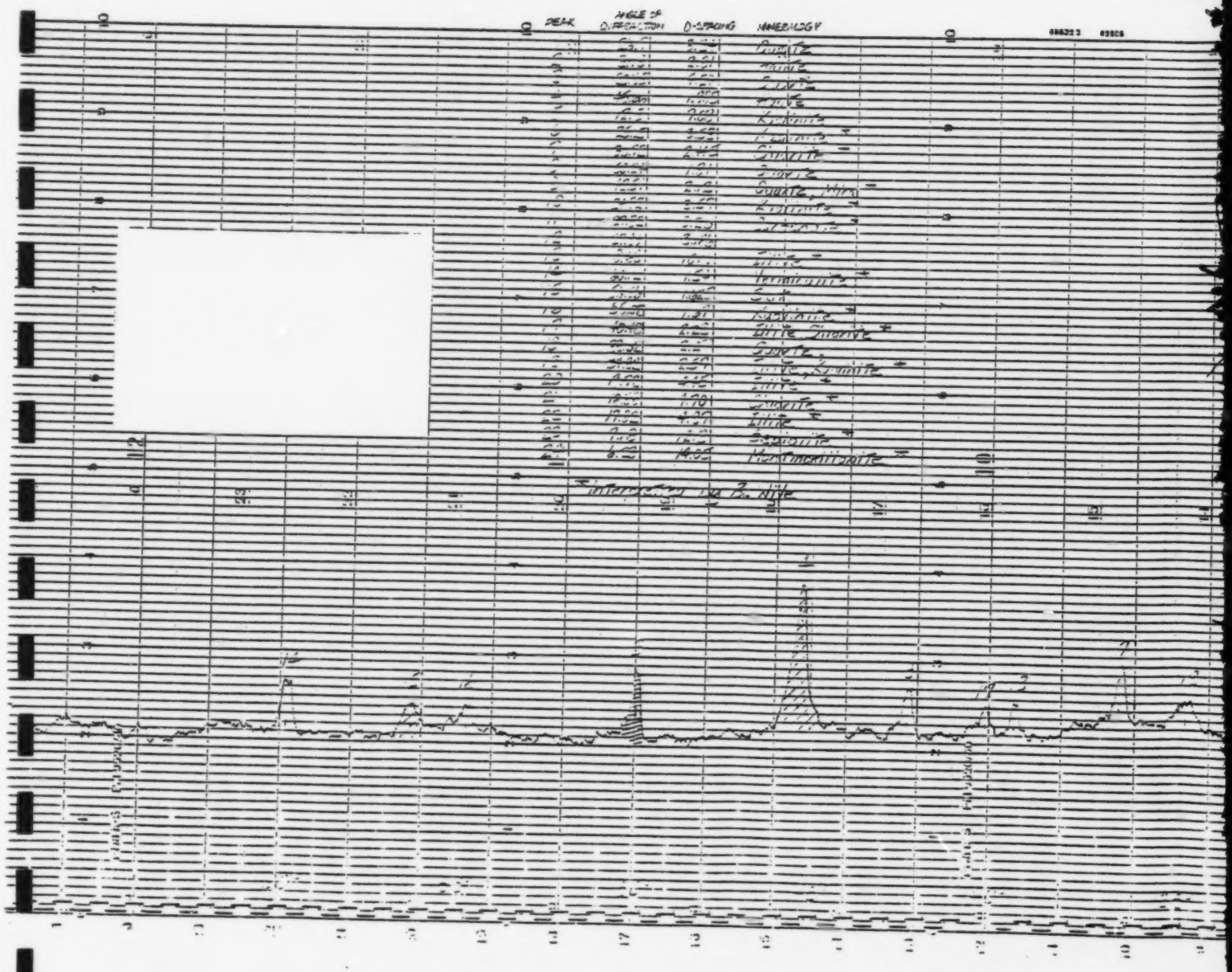


FIGURE 30 : X-RAY DIFFRACTOGRAMS OF LINGAN 9E-TOP SHALE


PEAK
ANGLE OF DIFFRACTION
D-SPACING
MINERALOGY

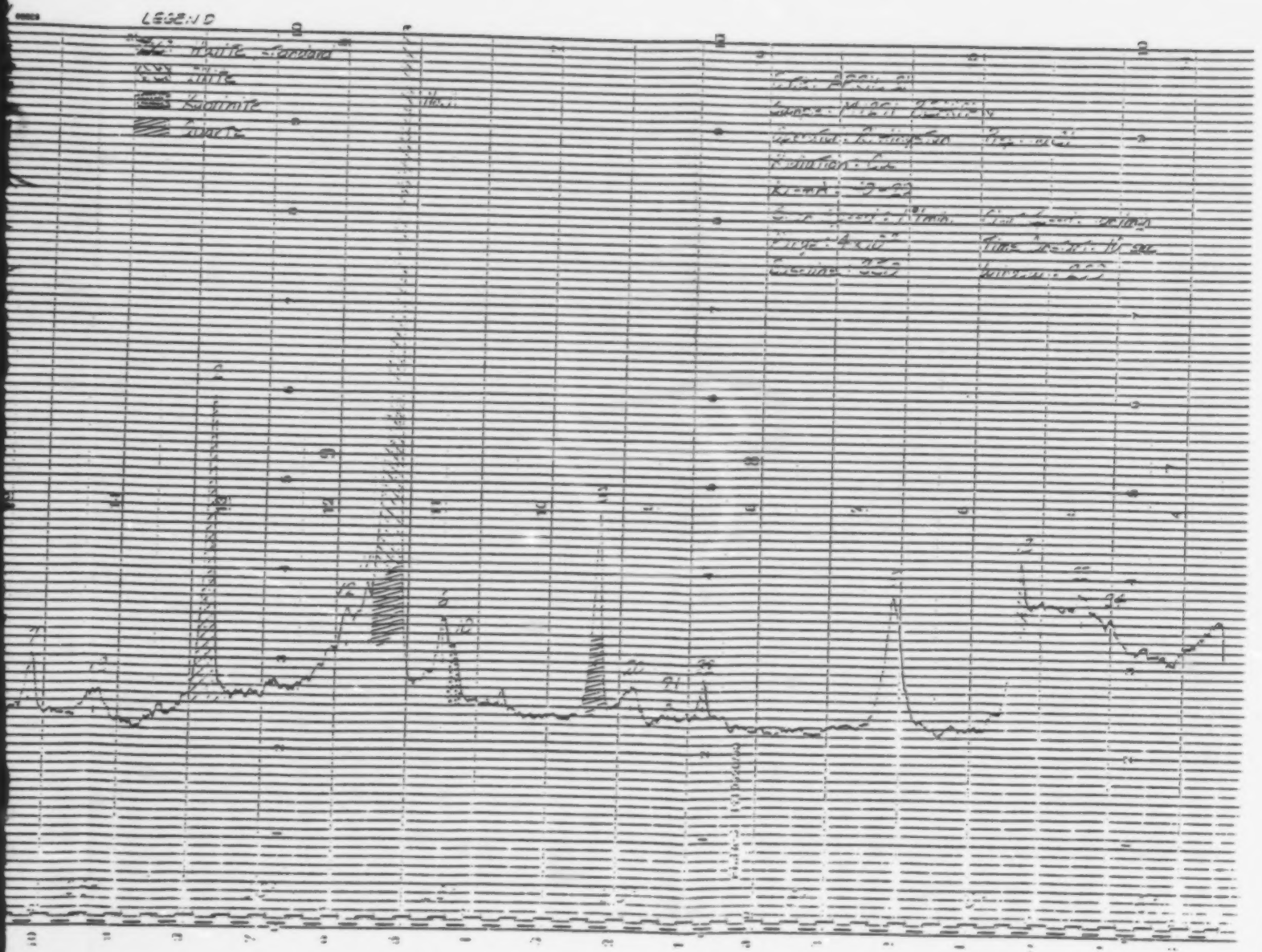


FIGURE 31: X-RAY DIFFRACTOGRAMS OF BEARPAW SHALE

BOREHOLE	SAMPLE	DEPTH
12	1	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
14.0000	0.0	0.0	0.0	0.0	100.00
10.0000	1.4	0.0	0.0	0.0	96.03
5.0000	10.9	0.0	0.0	0.0	65.16
2.5000	12.6	0.0	0.0	0.0	29.46
1.2500	5.2	0.0	0.0	0.0	14.77
.6300	1.5	0.0	0.0	0.0	10.48
.3150	.5	0.0	0.0	0.0	9.07
.1e00	.1	0.0	0.0	0.0	8.78
.0800	0.0	0.0	0.0	0.0	8.78

Sample weight for sieve analysis = 35.3 grams.

Note: DATA FOR EVANS SAMPLE IN NORMAL TAP WATER

BOREHOLE	SAMPLE	DEPTH
12	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	6.5	0.0	0.0	0.0	89.78
10.0000	19.2	0.0	0.0	0.0	59.39
5.0000	27.3	0.0	0.0	0.0	16.87
2.5000	7.1	0.0	0.0	0.0	6.89
1.2500	1.9	0.0	0.0	0.0	4.22
.6300	.6	0.0	0.0	0.0	3.37
.3150	.2	0.0	0.0	0.0	3.09
.1e00	.1	0.0	0.0	0.0	2.95
.0800	0.0	0.0	0.0	0.0	2.95

Sample weight for sieve analysis = 67.6 grams.

Note: DATA FOR EVANS SAMPLE IN KCL SOLUTION

BOREHOLE	SAMPLE	DEPTH
12	3	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	4.6	0.0	0.0	0.0	92.07
10.0000	6.2	0.0	0.0	0.0	81.08
5.0000	22.9	0.0	0.0	0.0	40.16
2.5000	12.6	0.0	0.0	0.0	17.26
1.2500	4.4	0.0	0.0	0.0	9.67
.6300	1.2	0.0	0.0	0.0	7.50
.3150	.5	0.0	0.0	0.0	6.72
.1e00	.2	0.0	0.0	0.0	6.40
.0800	.1	0.0	0.0	0.0	6.22

Sample weight for sieve analysis = 58.0 grams.

Note: DATA FOR EVANS SAMPLE IN NACL SOLUTION

Grain Size Distribution Data for Evans Shale in Normal Tap Water, KCl Solution and Sodium Chloride Solution (NaCl).

BOREHOLE 15 SAMPLE 11 DEPTH

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	25.8	0.0	0.0	0.0	51.78
10.0000	5.4	0.0	0.0	0.0	41.04
5.0000	8.6	0.0	0.0	0.0	28.94
2.5000	1.4	0.0	0.0	0.0	26.27
1.2500	1.8	0.0	0.0	0.0	22.91
.6300	1.2	0.0	0.0	0.0	20.76
.3150	.9	0.0	0.0	0.0	19.08
.1600	.6	0.0	0.0	0.0	17.94
.0800	.3	0.0	0.0	0.0	17.40

Sample weight for sieve analysis = 50.3 grams.

Note: BEARPAW SHALE IN NORMAL TAP WATER

BOREHOLE 15 SAMPLE 12 DEPTH

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
40.0000	0.0	0.0	0.0	0.0	100.00
28.0000	32.0	0.0	0.0	0.0	14.89
20.0000	0.0	0.0	0.0	0.0	14.89
14.0000	3.2	0.0	0.0	0.0	6.38
10.0000	1.7	0.0	0.0	0.0	1.86
5.0000	.4	0.0	0.0	0.0	.80
2.5000	.1	0.0	0.0	0.0	.53
1.2500	.1	0.0	0.0	0.0	.27
.6300	.1	0.0	0.0	0.0	0.00

Sample weight for sieve analysis = 37.6 grams.

Note: BEARPAW SHALE IN KCL SOLUTION

BOREHOLE 15 SAMPLE 13 DEPTH

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
28.0000	0.0	0.0	0.0	0.0	100.00
20.0000	20.0	0.0	0.0	0.0	44.43
14.0000	5.9	0.0	0.0	0.0	28.04
10.0000	3.1	0.0	0.0	0.0	8.31
5.0000	1.5	0.0	0.0	0.0	4.14
2.5000	.6	0.0	0.0	0.0	2.47
1.2500	.1	0.0	0.0	0.0	2.20
.6300	.1	0.0	0.0	0.0	1.92
.3150	.1	0.0	0.0	0.0	1.64

Sample weight for sieve analysis = 36.0 grams.

Note: BEARPAW SHALE IN NACL SOLUTION

Grain Size Distribution Data for Bearpaw Shale in Normal Tap Water, KCl Solution and NaCl Solution.

BOREHOLE	SAMPLE	DEPTH
23	1	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	2.9	0.0	0.0	0.0	95.92
10.0000	7.1	0.0	0.0	0.0	85.92
5.0000	32.3	0.0	0.0	0.0	40.42
2.5000	19.8	0.0	0.0	0.0	12.54
1.2500	5.9	0.0	0.0	0.0	4.23
.6300	1.5	0.0	0.0	0.0	2.11
.3150	.6	0.0	0.0	0.0	1.27
.1600	.3	0.0	0.0	0.0	.85
.0800	.1	0.0	0.0	0.0	.70

Sample weight for sieve analysis = 71.0 grams.

Note: EVANS SHALE IN 3% KCL & SS-100 POLYMER

BOREHOLE	SAMPLE	DEPTH
23	3	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
28.0000	0.0	0.0	0.0	0.0	100.00
20.0000	17.6	0.0	0.0	0.0	80.27
14.0000	9.3	0.0	0.0	0.0	69.84
10.0000	13.6	0.0	0.0	0.0	54.60
5.0000	27.8	0.0	0.0	0.0	23.43
2.5000	13.1	0.0	0.0	0.0	8.74
1.2500	4.6	0.0	0.0	0.0	3.59
.6300	1.6	0.0	0.0	0.0	1.79
.3150	.7	0.0	0.0	0.0	1.01
.1600	.3	0.0	0.0	0.0	.67
.0800	.1	0.0	0.0	0.0	.56

Sample weight for sieve analysis = 89.2 grams.

Note: EVANS SHALE IN 5.4 % KCL & SS-100 POLYMER

BOREHOLE	SAMPLE	DEPTH
23	4	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	4.3	0.0	0.0	0.0	96.24
10.0000	10.0	0.0	0.0	0.0	87.50
5.0000	37.7	0.0	0.0	0.0	37.06
2.5000	27.2	0.0	0.0	0.0	13.29
1.2500	9.0	0.0	0.0	0.0	5.42
.6300	3.1	0.0	0.0	0.0	2.71
.3150	1.5	0.0	0.0	0.0	1.40
.1600	.6	0.0	0.0	0.0	.87
.0800	.2	0.0	0.0	0.0	.70

Sample weight for sieve analysis = 114.4 grams.

Note: EVANS SHALE IN 7.89 % KCL & SS-100 POLYMER

Grain Size Distribution Data for Evans Shale in KCl and SS-100 Polymer Solutions.

BOREHOLE	SAMPLE	DEPTH
24	3	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
10.0000	0.0	0.0	0.0	0.0	100.00
5.0000	19.0	0.0	0.0	0.0	71.34
2.5000	26.8	0.0	0.0	0.0	30.92
1.2500	12.1	0.0	0.0	0.0	12.67
.6300	4.7	0.0	0.0	0.0	5.58
.3150	2.3	0.0	0.0	0.0	2.11
.1600	1.0	0.0	0.0	0.0	.60
.0800	.4	0.0	0.0	0.0	-0.00

Sample weight for sieve analysis = 66.3 grams.

Note: EVANS SHALE IN 3% KCL AND ALCOMER POLYMER

BOREHOLE	SAMPLE	DEPTH
24	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
28.0000	0.0	0.0	0.0	0.0	100.00
20.0000	30.4	0.0	0.0	0.0	74.83
14.0000	26.6	0.0	0.0	0.0	52.81
10.0000	8.1	0.0	0.0	0.0	46.11
5.0000	29.3	0.0	0.0	0.0	21.85
2.5000	18.8	0.0	0.0	0.0	6.29
1.2500	5.2	0.0	0.0	0.0	1.99
.6300	1.6	0.0	0.0	0.0	.66
.3150	.6	0.0	0.0	0.0	.17
.1600	.2	0.0	0.0	0.0	0.00
.0800	0.0	0.0	0.0	0.0	0.00

Sample weight for sieve analysis = 120.8 grams.

Note: EVANS SAMPLE IN 5.4% KCL AND ALCOMER POLYMER

BOREHOLE	SAMPLE	DEPTH
24	1	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
14.0000	0.0	0.0	0.0	0.0	100.00
10.0000	6.6	0.0	0.0	0.0	94.59
5.0000	55.1	0.0	0.0	0.0	49.38
2.5000	41.2	0.0	0.0	0.0	15.38
1.2500	9.2	0.0	0.0	0.0	8.03
.6300	2.3	0.0	0.0	0.0	6.14
.3150	.9	0.0	0.0	0.0	5.41
.1600	.4	0.0	0.0	0.0	5.08
.0800	.1	0.0	0.0	0.0	5.00

Sample weight for sieve analysis = 121.9 grams.

Note: EVANS SHALE IN 7.9% KCL AND ALCOMER POLYMER

Grain Size Distribution Data for Evans Sample in KCl and Alcomer Polymer Solutions.

BOREHOLE	SAMPLE	DEPTH
24	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
28.0000	0.0	0.0	0.0	0.0	100.00
20.0000	30.4	0.0	0.0	0.0	74.83
14.0000	26.6	0.0	0.0	0.0	52.81
10.0000	8.1	0.0	0.0	0.0	46.11
5.0000	29.3	0.0	0.0	0.0	21.85
2.3000	18.8	0.0	0.0	0.0	6.29
1.2300	5.2	0.0	0.0	0.0	1.99
.6300	1.6	0.0	0.0	0.0	.66
.3150	.6	0.0	0.0	0.0	.17
.1600	.2	0.0	0.0	0.0	0.00
.0800	0.0	0.0	0.0	0.0	0.00

Sample weight for sieve analysis = 120.8 grams.

Note: EVANS SHALE IN 5.4% KCL AND ALCOMER

BOREHOLE	SAMPLE	DEPTH
23	3	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
28.0000	0.0	0.0	0.0	0.0	100.00
20.0000	17.6	0.0	0.0	0.0	80.27
14.0000	9.3	0.0	0.0	0.0	69.84
10.0000	13.6	0.0	0.0	0.0	54.60
5.0000	27.8	0.0	0.0	0.0	23.43
2.3000	13.1	0.0	0.0	0.0	8.74
1.2300	4.6	0.0	0.0	0.0	3.59
.6300	1.6	0.0	0.0	0.0	1.79
.3150	.7	0.0	0.0	0.0	1.01
.1600	.3	0.0	0.0	0.0	.67
.0800	.1	0.0	0.0	0.0	.56

Sample weight for sieve analysis = 89.2 grams.

Note: EVANS SHALE IN 5.4% KCL AND SS-100 POLYMER

BOREHOLE	SAMPLE	DEPTH
12	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	6.3	0.0	0.0	0.0	89.78
10.0000	19.2	0.0	0.0	0.0	59.59
5.0000	27.3	0.0	0.0	0.0	16.67
2.3000	7.1	0.0	0.0	0.0	5.50
1.2300	1.9	0.0	0.0	0.0	2.52
.6300	.6	0.0	0.0	0.0	1.57
.3150	.2	0.0	0.0	0.0	1.26
.1600	.1	0.0	0.0	0.0	1.10
.0800	0.0	0.0	0.0	0.0	1.10

Sample weight for sieve analysis = 63.6 grams.

Note: EVANS SHALE IN 7.9 % KCL SOLUTION.

Grain Size Distribution Data of Evans Shale in 5.4% KCl Soultion and Alcomer Polymer, 5.4% KCl and SS-100 Polymer and 7.9% KCl Solution.

BOREHOLE	SAMPLE	DEPTH
13	12	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
40.0000	0.0	0.0	0.0	0.0	100.00
28.0000	32.0	0.0	0.0	0.0	14.89
20.0000	0.0	0.0	0.0	0.0	14.89
14.0000	3.2	0.0	0.0	0.0	6.38
10.0000	1.7	0.0	0.0	0.0	1.86
5.0000	.4	0.0	0.0	0.0	.80
2.5000	.1	0.0	0.0	0.0	.53
1.2500	.1	0.0	0.0	0.0	.27
.6300	.1	0.0	0.0	0.0	0.00

Sample weight for sieve analysis = 37.6 grams.

Note: BEARPAW SHALE IN 7.9% KCL SOLUTION

BOREHOLE	SAMPLE	DEPTH
24	4	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
40.0000	0.0	0.0	0.0	0.0	100.00
28.0000	29.6	0.0	0.0	0.0	40.08
14.0000	6.7	0.0	0.0	0.0	26.32
10.0000	6.3	0.0	0.0	0.0	13.77
5.0000	5.0	0.0	0.0	0.0	3.64
2.5000	.8	0.0	0.0	0.0	2.02
1.2500	.6	0.0	0.0	0.0	.81
.6300	.3	0.0	0.0	0.0	.20
.3150	.1	0.0	0.0	0.0	-0.00
.1600	0.0	0.0	0.0	0.0	-0.00

Sample weight for sieve analysis = 49.4 grams.

BOREHOLE	SAMPLE	DEPTH
23	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
40.0000	0.0	0.0	0.0	0.0	100.00
28.0000	34.0	0.0	0.0	0.0	11.23
14.0000	2.8	0.0	0.0	0.0	3.92
10.0000	1.4	0.0	0.0	0.0	.26
5.0000	.1	0.0	0.0	0.0	0.00

Sample weight for sieve analysis = 38.3 grams.

Grain Size Distribution Data for Bearpaw Shale in 3% KCl and Alcomer Polymer, 3% MCl and SS-100 Polymer, and 7.9% KCl Solution.

BOREHOLE	SAMPLE	DEPTH
24	6	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
10.0000	0.0	0.0	0.0	0.0	100.00
5.0000	17.4	0.0	0.0	0.0	80.92
2.5000	28.9	0.0	0.0	0.0	49.22
1.2500	22.9	0.0	0.0	0.0	23.01
.6300	8.4	0.0	0.0	0.0	12.90
.3150	4.3	0.0	0.0	0.0	9.08
.1600	2.0	0.0	0.0	0.0	6.89
.0800	.9	0.0	0.0	0.0	5.90

Sample weight for sieve analysis = 91.2 grams.

Note: EVANS SHALE IN 4.76% CaCl SOLUTION

BOREHOLE	SAMPLE	DEPTH
12	1	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
14.0000	0.0	0.0	0.0	0.0	100.00
10.0000	1.4	0.0	0.0	0.0	96.03
5.0000	10.9	0.0	0.0	0.0	65.16
2.5000	12.6	0.0	0.0	0.0	29.46
1.2500	5.2	0.0	0.0	0.0	14.73
.6300	1.5	0.0	0.0	0.0	10.48
.3150	.5	0.0	0.0	0.0	9.07
.1600	.1	0.0	0.0	0.0	8.78
.0800	0.0	0.0	0.0	0.0	8.78

Sample weight for sieve analysis = 35.3 grams.

Note: DATA FOR EVANS SAMPLE IN NORMAL TAP WATER

BOREHOLE	SAMPLE	DEPTH
12	2	

SIEVE SIZE (mm)	WEIGHT RETAINED (grams)	TIME (min)	TEMP (deg C)	HYD RDG	PERCENT PASSING (plotted)
20.0000	0.0	0.0	0.0	0.0	100.00
14.0000	6.5	0.0	0.0	0.0	89.78
10.0000	19.2	0.0	0.0	0.0	59.39
5.0000	27.3	0.0	0.0	0.0	16.67
2.5000	7.1	0.0	0.0	0.0	5.50
1.2500	1.9	0.0	0.0	0.0	2.52
.6300	.6	0.0	0.0	0.0	1.67
.3150	.2	0.0	0.0	0.0	1.26
.1600	.1	0.0	0.0	0.0	1.10
.0800	0.0	0.0	0.0	0.0	1.10

Sample weight for sieve analysis = 63.6 grams.

Note: DATA FOR EVANS SAMPLE IN KCL SOLUTION

Grain Size Distribution Data for Evans Shale in 4.76% $Cacl_2$ Solution.

APPENDIX C

STANDARD TEST PROCEDURES

C-1	CANMET REPORT 77-25,	DETERMINATION OF WATER CONTENT
C-2	CANMET REPORT 77-25,	DETERMINATION OF POROSITY/ DENSITY
C-3	ASTM D423-66	STANDARD TEST METHOD FOR LIQUID LIMIT OF SOILS
C-4	ASTM D424-59	STANDARD TEST METHOD FOR PLASTIC LIMIT AND PLASTIC INDEX OF SOILS
C-5	ASTM D422-63	STANDARD TEST FOR PARTICLE SIZE ANALYSIS OF SOILS
C-6	CANMET REPORT 77-25,	DETERMINATION OF SLAKE-DURABILITY INDEX
C-7	X-RAY ANALYSIS,	AS SUBCONTRACTED TO CUESTA RESEARCH LIMITED
C-8	ADSORPTION ISOTHERM TEST	AFTER O'BRIEN AND CHENEVERT SEPTEMBER 1973

APPENDIX C-1

DETERMINATION OF WATER CONTENT

SCOPE

8. The purpose of this test is to measure the weight of water contained in a rock sample, as a percentage of the oven-dry sample weight.

APPARATUS

9. a. an oven capable of maintaining a temperature of 105° C to within 3° C for a period of at least 24 hours

b. sample containers of non-corrodible material, including airtight lids

c. a desiccator to hold sample containers during cooling

d. a balance of adequate capacity, capable of weighing to an accuracy of 0.01 g.

PROCEDURE

10. a. The containers and lids are cleaned and dried and their weights, A, measured.

b. A representative sample is selected, preferably comprising at least five rock lump specimens, each weighing at least 50 g.

c. The specimens are placed in the containers and the lids replaced.

d. The weight, B, of each specimen together with container and lid is determined.

e. The lids are removed and specimens dried to a constant weight at a temperature of 105° C.

f. The lids are replaced and the specimens allowed to cool in the desiccator for 30 minutes.

g. The weight, C, of each specimen together with container and lid is measured.

11. To retain the original water content of the sample the time gap between steps 10(c) and 10(d) should be as brief as possible. Guidance for storing and handling samples is given in Supplement 3-5.

CALCULATION

12. Water content, $w\% = (W_w/W_s)\ 100$

where pore water weight, $W_w = B-C$

solid weight, $W_s = C-A$

REPORTING OF RESULTS

13. The water content should be reported to the nearest 0.1% in tabulated form with proper sample identification (rock type and origin) if being determined for several locations (eg the water content at various depths of a borehole or various sections and/or elevations of the pit wall).

14. The working sheets are adequate substitutes for a report if only a few sample locations are involved in the water content determination (Fig 1).

WATER CONTENT

Sample altered carbonate of the shear zone

Location 5th bench, toe elevation Date July 16, 1973

Bore Hole _____ Depth _____ Tested by W G

Sample No. North fault 5

Specimen No.	Container No.	Tare A	Wet B	Dry C	W_w	W_s	w%
F-21	52	13.01	65.91	61.28	4.63	48.27	9.6
F-22	47	12.98	68.91	64.34	4.57	51.36	8.9
F-23	16	12.79	72.85	67.99	4.86	55.20	8.8
F-24	29	12.86	62.99	58.81	4.18	45.95	9.1
F-25	5	12.95	71.24	66.43	4.81	53.48	9.0

Sample water content: 9 %

Remarks: specimens were placed into containers at the site on July 15, kept in lab. until testing covered with damp rag.

Fig 1 - Working sheet for water content determination.

APPENDIX C-2

DETERMINATION OF POROSITY/DENSITY

SCOPE

15. a. The purpose of this test is to measure porosity, dry density and related properties of a rock sample.

b. The method applies equally well to regularly shaped rock specimens, and rock samples in the form of lumps or aggregates of irregular shape.

c. The method can be applied to coherent rock materials as well as to those which may swell or disintegrate if immersed in water.

d. All procedures for determining porosity/density are based on the appropriate standards (1,3).

APPARATUS

16. a. an oven capable of maintaining a temperature of 105° C within 3° C for a period of at least 24 hours

b. specimen containers of non-corrodible material, including airtight lids

c. a desiccator to hold specimen containers during cooling

d. a balance of adequate capacity, capable of weighing to an accuracy of 0.01 g

e. evaporating dishes - two large and one smaller

f. a glass plate large enough to cover the small dish

g. a graduated cylinder (conveniently 200 cm^3)

h. a vacuum apparatus such as a vacuum pump or water faucet aspirator providing vacuum less than 0.1 psi (0.7 kPa)

i. grinding equipment to reduce the sample to a powder less than 0.42 mm (40 sieve) in grain size

j. a calibrated volumetric flask and stopper (conveniently 50 cm^3).

PROCEDURE

17. a. The containers and lids are cleaned and dried, and their weights, A, measured.

b. A representative sample is selected, preferably comprising at least five rock specimens. The shape and size of the pieces should suit the capacity of the measuring apparatus.

c. The specimens are placed in the containers and the lids replaced.

d. The weight, B, of each specimen container with lid is determined.

e. The lids are removed and the specimens dried to constant weight at a temperature of 105° C.

f. The lids are replaced and the specimens allowed to cool in the desiccator for 30 minutes.

g. The weight, C, of each specimen together with container and lid is measured.

18. a. A loop of thread is tied around each specimen so that approximately three inches extend beyond the knot.

b. Each specimen is dipped into melted paraffin by holding the end of the thread and then held in air for a few seconds until the paraffin coating hardens. The excess length of thread beyond the knot is removed and the specimen replaced in its container.

c. The weight, D, of the paraffin-coated specimens plus containers and lids is measured.

19. a. The small dish, placed in a larger one, is filled to overflowing with water, then covered with the glass plate so that the plate is flush with the top of the dish and no air is entrapped.

b. The outside of the small dish is wiped clean and placed in the second larger dish, which is clean and empty.

c. The glass plate is removed and the paraffin-coated specimen is submerged; the dish is then covered with the glass plate which is again placed flush with its upper surface. The small dish is removed from the larger one, allowing the water from its outside surface to drip back into the larger dish.

d. The water displaced by the paraffin-coated specimen is poured into a clean, empty graduated cylinder and its volume, E, recorded.

20. If a drill core specimen of cylindrical shape is used the bulk volume is calculated as follows:

a. The diameter, d, of the specimen is measured to the nearest 0.005 in. (0.1 mm) by averaging two diameters measured at right angles to each other at about the upper height, mid height and lower height of the specimen. The diameter used to calculate volume is then the average of the three diameters obtained at the upper height, mid height and lower height of the specimen.

b. Specimen height, L_o, is determined to the nearest 0.005 in. (0.1 mm) by averaging the four heights measured along the side of the specimen, at two diameters at right angles to each other.

21. a. The paraffin coating is removed from the specimens by scraping and chipping and the cleaned specimens are crushed together and ground to a grain size not exceeding 0.42 mm (40 sieve). A representative sub-sample of about 15 g of the pulverized material is selected and oven dried.

b. The weight, F, of a clean and dry volumetric flask together with stopper is measured.

c. The flask is filled with distilled water, then brought to an equilibrium temperature by keeping it at uniform room temperature for three hours. Its water level is adjusted accurately to the 50 cm^3 graduation mark with a medicine dropper. The flask is sealed with a stopper and its weight, G, measured.

d. The flask is emptied and dried and a 15 g sample of dry, pulverized rock added with the aid of a funnel. The weight, H, of the flask including sample and stopper is measured.

e. Sufficient distilled water is added to thoroughly wet the sample; the air from the flask and wet sample is then evacuated for about 20 minutes. Further distilled water is added, and the flask again carefully evacuated to remove air. Finally the water level is accurately adjusted to the 50 cm^3 graduation mark.

f. The stoppered flask with contents is kept for three hours at a uniform room temperature when its weight, J, is measured.

CALCULATIONS

22. a. Water content, $w\% = (W_w/W_s)\ 100$

where pore water weight, $W_w = B-C$

solid weight, $W_s = C-A$

b. Density of rock, $\gamma = W_b/V_b$

where bulk weight, $W_b = B-A$

bulk volume, $V_b = E-(D-C)/\gamma_p$

density of paraffin, $\gamma_p = 0.908\ g/cm^3$

or

bulk volume, $V_b = d^2\ \pi\ L_o/4$

where d = diameter of the cylindrical specimen

L_o = height of the cylindrical specimen,

c. Dry density of rock, $\gamma_d = W_s/V_b$

d. Grain density, $\gamma_g = G_w/G_v$

where grain weight, $G_w = H-F$

grain volume, $G_v = V[1-(J-H)/(G-F)]$

V = calibrated volume of flask, usually 50 cm^3

e. Porosity, $n\% = 100\ (1-\gamma_d/\gamma_g)$

f. Degree of saturation,

$$S_r\% = 100\ w\gamma_d/n\gamma_w$$

where density of water,

γ_w = 1.00 g/cm^3 or 62.4 lb/ft^3

REPORTING OF RESULTS

23. If the porosity/density and related properties are being determined for several locations (eg at various depths of a borehole or various sections and/or elevations of the pit wall) the results are reported in tabulated form, with proper sample identification (rock type and origin) and with accuracies indicated in Fig 2.

24. If the tests involve only a few samples the working sheets as shown in Fig 2 substitute adequately for a report.

POROSITY / DENSITY

Sample *weathered limestone*

Location *footwall outcrop, K zone* Date *July 23, 1973*

Bore Hole _____ Depth _____ Tested by *W.G.*

Sample No. *K*

Specimen No.	Container No.	A	B	C	D	E	D-C	$\frac{D-C}{\gamma_p}$	W_w	W_s	W_b	V_b	w%	γ	γ_d
K-6	16	12.79	80.20	75.91	80.46	36.5	4.55	5.01	4.29	63.12	67.41	30.99	6.8	2.17	2.04
K-7	5	12.95	89.53	84.19	91.17	42.0	6.98	7.69	5.34	71.24	76.58	34.31	7.5	2.23	2.08
K-8	38	13.16	78.80	74.11	79.56	37.5	5.45	6.00	4.69	60.95	65.64	31.50	7.7	2.08	1.93
K-9	14	13.05	74.99	71.21	76.07	32.5	4.86	5.35	3.78	58.16	61.94	27.15	6.5	2.28	2.14
K-10	43	12.88	76.47	71.98	77.14	34.0	5.16	5.68	4.49	59.10	63.59	28.32	7.6	2.24	2.09

F: *42.68*

G: *92.59*

H: *57.96*

J: *102.18*

$G_w = H - F =$ *15.28*

$G_v = V\left[1-(J-H)/(G-F)\right] =$ *5.70*

$\gamma_g = G_w/G_v =$ *2.68*

Sample water content, w: *7.2* %

bulk density, γ: *2.20* g/cm^3, *137* lb/ft^3

dry density, γ_d: *2.06* g/cm^3, *128* lb/ft^3

grains density, γ_g: *2.68* g/cm^3, *167* lb/ft^3

porosity, n: *23.1* %

degree of saturation, S_r: *64.2* %

Remarks: *specimens were collected on July 19, placed into containers, covered with damp rag in lab. until testing.*

Fig 2 - Working sheet for porosity/density determination.

AMERICAN NATIONAL STANDARD ANSI/ASTM D 423 - 66 (Reapproved 1972)

APPENDIX C-3

Standard Test Method for LIQUID LIMIT OF SOILS[1]

This Standard is issued under the fixed designation D 423; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the determination of the liquid limit of soils as defined in Section 2, using the mechanical device specified in Section 3, securing the results of at least three trials, and the plotting of a flow curve. Provision is also made for: (*1*) a one-point method requiring the calculation of the liquid limit value from data secured from a single trial; and (*2*) a procedure for check or referee tests.

1.2 The mechanical method shall be used unless specifications for the soil being tested permit the use of the one-point method. The procedure for check or referee tests shall be used in cases of disagreement or when required in the specifications for the soil being tested.

2. Definition

2.1 *liquid limit of soil*—the water content, expressed as a percentage of the weight of the oven-dried soil, at the boundary between the liquid and plastic states. The water content at this boundary is arbitrarily defined as the water content at which two halves of a soil cake will flow together for a distance of ½ in. (12.7 mm) along the bottom of the groove separating the two halves, when the cup is dropped 25 times for a distance of 1 cm (0.3937 in.) at the rate of 2 drops/s.

MECHANICAL METHOD

3. Apparatus

3.1 *Evaporating Dish*—A porcelain evaporating dish about 4½ in. (114.3 mm) in diameter.

3.2 *Spatula*—A spatula or pill knife having a blade about 3 in. (76.2 mm) in length and about ¾ in. (19.0 mm) in width.

3.3 *Liquid Limit Device*—A mechanical device consisting of a brass cup and carriage, constructed according to the plan and dimensions shown in Fig. 1.[2]

3.4 *Grooving Tool*—A combined grooving tool and gage conforming to the dimensions shown in Fig. 1.

3.5 *Containers*—Suitable containers, such as matched watch glasses, that will prevent loss of moisture during weighing.

3.6 *Balance*—A balance sensitive to 0.01 g.

4. Sample

4.1 Take a sample weighing about 100 g from the thoroughly mixed portion of the material passing the No. 40 (425-μm) sieve, obtained in accordance with ASTM Method D 421, Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants.[3]

5. Adjustment of Mechanical Device

5.1 Inspect the liquid-limit device to determine that the device is in good working order, that the pin connecting the cup is not worn sufficiently to permit side play, that the screws connecting the cup to the hanger arm are tight, and that a groove has not been worn in the cup through long usage. Inspect the

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock for Engineering Purposes.
Current edition approved Sept. 20, 1966. Originally issued 1935. Replaces D 423 - 61.

[2] Detailed working drawings for this liquid limit device are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103. Order Adjunct No. 12-404230-00.

[3] *Annual Book of ASTM Standards*, Part 19.

grooving tool to determine that the critical dimensions are as shown in Fig. 1.

5.2 By means of the gage on the handle of the grooving tool and the adjustment plate *H*, Fig. 1, adjust the height to which the cup *C* is lifted so that the point on the cup that comes in contact with the base is exactly 1 cm (0.3937 in.) above the base. Secure the adjustment plate *H* by tightening the screws, *I*. With the gage still in place, check the adjustment by revolving the crank rapidly several times. If the adjustment is correct, a slight ringing sound will be heard when the cam strikes the cam follower. If the cup is raised off the gage or no sound is heard, make further adjustments.

6. Procedure

6.1 Place the soil sample in the evaporating dish and thoroughly mix with 15 to 20 ml of distilled water by alternately and repeatedly stirring, kneading, and chopping with a spatula. Make further additions of water in increments of 1 to 3 ml. Thoroughly mix each increment of water with the soil, as previously described, before adding another increment of water.

6.2 When sufficient water has been thoroughly mixed with the soil to produce a consistency that will require 30 to 35 drops of the cup to cause closure, place a portion of the mixture in the cup above the spot where the cup rests on the base, and squeeze it down and spread it into the position shown in Fig. 2 with as few strokes of the spatula as possible, care being taken to prevent the entrapment of air bubbles within the mass. With the spatula, level the soil and at the same time trim it to a depth of 1 cm at the point of maximum thickness. Return the excess soil to the evaporating dish. Divide the soil in the cup by firm strokes of the grooving tool along the diameter through the centerline of the cam follower so that a clean, sharp groove of the proper dimensions will be formed. To avoid tearing of the sides of the groove or slipping of the soil cake on the cup, up to six strokes, from front to back or from back to front counting as one stroke, shall be permitted. Each stroke should penetrate a little deeper until the last stroke from back to front scrapes the bottom of the cup clean. Make the groove with as few strokes as possible.

6.3 Lift and drop the cup by turning the crank, *F*, at the rate of 2 rps, until the two halves of the soil cake come in contact at the bottom of the groove along a distance of about ½ in. (12.7 mm). Record the number of drops required to close the groove along a distance of ½ in.

6.4 Remove a slice of soil approximately the width of the spatula, extending from edge to edge of the soil cake at right angles to the groove and including that portion of the groove in which the soil flowed together, and place in a suitable tared container. Weigh and record the mass. Oven-dry the soil in the container to constant mass at 230 ± 9 F (110 ± 5 C) and reweigh as soon as it has cooled but before hygroscopic moisture can be absorbed. Record this mass. Record the loss in mass due to drying as the mass of water.

6.5 Transfer the soil remaining in the cup to the evaporating dish. Wash and dry the cup and grooving tool, and reattach the cup to the carriage in preparation for the next trial.

6.6 Repeat the foregoing operations for at least two additional trials, with the soil collected in the evaporating dish, to which sufficient water has been added to bring the soil to a more fluid condition. The object of this procedure is to obtain samples of such consistency that the number of drops required to close the groove will be above and below 25. The number of drops should be less than 35 and exceed 15. The test shall always proceed from the dryer to the wetter condition of the soil.

7. Calculation

7.1 Calculate the water content w_N of the soil, expressed as a percentage of the weight of the oven-dried soil, as follows:

$$w_N = (\text{mass of water}/\text{mass of oven-dried soil}) \times 100$$

8. Preparation of the Flow Curve

8.1 Plot a "flow curve" representing the relationship between water content and corresponding numbers of drops of the cup on a semilogarithmic graph with the water content as abscissae on the arithmetical scale, and the number of drops as ordinates on the logarithmic scale. The flow curve is a straight line drawn as nearly as possible through the three or more plotted points.

9. Liquid Limit

9.1 Take the water content corresponding to the intersection of the flow curve with the 25-drop ordinate as the liquid limit of the soil. Report this value to the nearest whole number.

ONE-POINT METHOD

10. Apparatus

10.1 The requirements for apparatus are the same as specified in Section 3.

11. Sample

11.1 The requirements for the sample are the same as specified in Section 4.

12. Adjustment of Mechanical Device

12.1 The requirements for the mechanical device are the same as specified in Section 5.

13. Procedure

13.1 Proceed in accordance with 6.1 through 6.5, except that a moisture content sample shall be taken only for the accepted trial. The accepted trial shall require between 20 and 30 drops of the cup to close the groove and at least two consistent consecutive closures shall be observed before taking the moisture-content sample for calculation of the liquid limit. The test shall always proceed from the dryer to the wetter condition of the soil.

14. Calculations

14.1 Calculate the water content w_N, for the accepted trial, expressed as a percentage of the oven-dried weight, as follows:

w_N = (mass of water/mass of oven-dried soil) × 100

14.2 Determine the liquid limit, LL, using the following formula:

$$LL = w_N(N/25)^{0.12}$$

where:

N = number of drops of the cup required to close the groove at the water content, w_N.

14.3 Report the liquid limit value to the nearest whole number. Values of $(N/25)^{0.12}$ are given in Table 1.

CHECK OR REFEREE TESTS

15. Factors Influencing Results

15.1 The results of liquid limit tests are influenced by:

15.1.1 The time required to make the test, and

15.1.2 The moisture content at which the test is begun.

16. Procedure

16.1 In making the liquid limit test for check or referee purposes, use the following time schedule:

16.1.1 Mixing of soil with water: 5 to 20 min, the longer period being used for the more plastic soils.

16.1.2 Seasoning in the humidifier: 24 h.

16.1.3 Remixing before placing in the brass cup: Add 1 ml of water and mix for 1 min. Add the water in 1-ml increments and remix until the soil appears to have such a consistency that the groove will close in 35 to 25 blows.

16.1.4 Placing in the brass cup, testing and weighing: 3 min.

16.1.5 Washing cup and grooving tool, adding water, and remixing: 3 min.

16.1.6 Repeat operations 4 and 5 for at least three satisfactory trials.

16.2 Do not record any trial requiring more than 35 or less than 15 drops of the cup. In no case shall dried soil be added to the seasoned soil being tested.

17. Report

17.1 The report shall include the following:

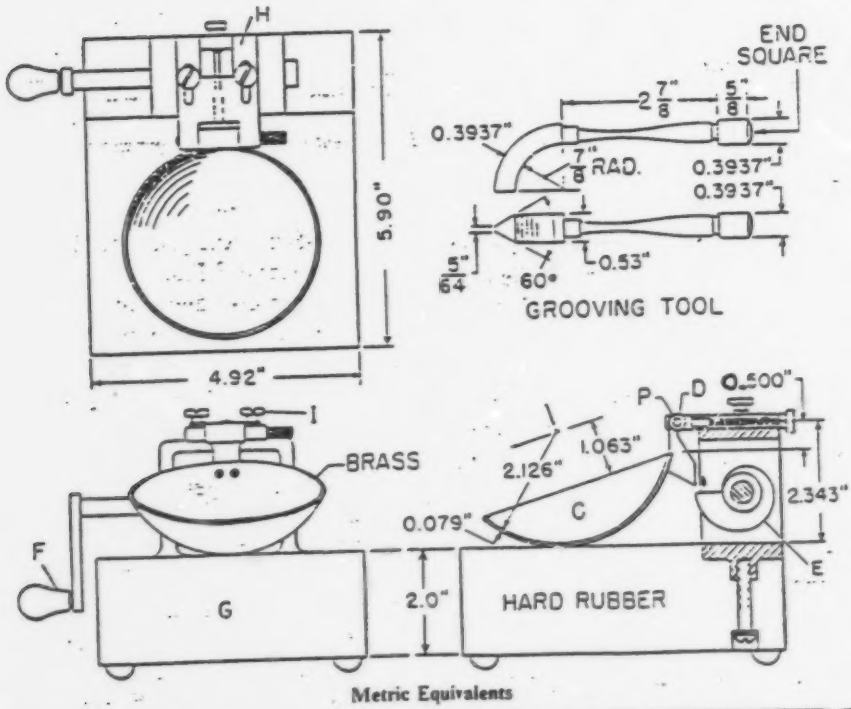
17.1.1 Liquid limit value, and

17.1.2 Procedure followed when either the one-point method or the procedure for check or referee tests is used.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103, which will schedule a further hearing regarding your comments. Failing satisfaction there, you may appeal to the ASTM Board of Directors.

TABLE 1 Values of $(N/25)^{0.12}$

N	$(N/25)^{0.12}$	N	$(N/25)^{0.12}$	N	$(N/25)^{0.12}$
20	0.974	24	0.995	28	1.014
21	0.979	25	1.000	29	1.018
22	0.985	26	1.005	30	1.022
23	0.990	27	1.009		



Metric Equivalents

in.	3/64	0.079	0.3937	0.500	0.53	5/8	7/8	1.063	2.0	2.126	2.343	2 7/8	4.92	5.90
mm	2.0	2.0	10.00	12.70	13.5	15.9	22.2	27.00	50	54.00	59.52	73.0	125.0	15.0

FIG. 1 Mechanical Liquid Limit Device.

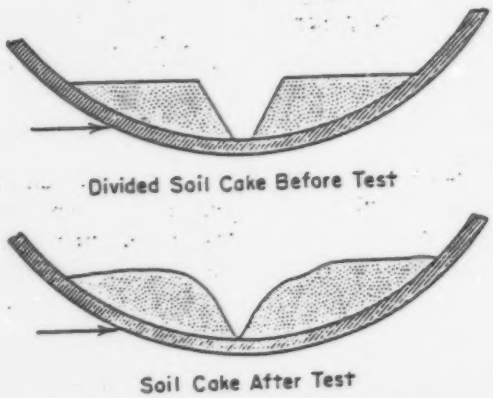


FIG. 2 Diagram Illustrating Liquid Limit Test.

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AMERICAN NATIONAL STANDARD ANSI/ASTM D 424 - 59 (Reapproved 1971)

APPENDIX C-4

Standard Test Method for
PLASTIC LIMIT AND PLASTICITY INDEX OF SOILS[1]

This Standard is issued under the fixed designation D 424; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 The plastic limit of a soil is the water content, expressed as a percentage of the mass of oven-dried soil, at the boundary between the plastic and semisolid states. The water content at this boundary is arbitrarily defined as the lowest water content at which the soil can be rolled into threads ⅛ in. (3.2 mm) in diameter without the threads breaking into pieces.

2. Apparatus

2.1 The apparatus shall consist of the following:

2.1.1 *Evaporating Dish*—A porcelain evaporating dish about 4½ in. (114.3 mm) in diameter.

2.1.2 *Spatula*—A spatula or pill knife having a blade about 3 in. (76.2 mm) in length and about ¾ in. (19.0 mm) in width.

2.1.3 *Surface for Rolling*—A ground-glass plate or piece of glazed or unglazed paper on which to roll the sample.

NOTE 1—"Unglazed" refers to paper similar to that used for mimeographing. Paper toweling is not satisfactory.

2.1.4 *Containers*—Suitable containers, such as matched watch glasses, that will prevent loss of moisture during weighing.

2.1.5 *Balance*—A balance sensitive to 0.01 g.

3. Sample

3.1 If the plastic limit only is required, take a quantity of soil weighing about 15 g from the thoroughly mixed portion of the material passing the No. 40 (425-μm) sieve, obtained in accordance with ASTM Method D 421, Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants.[2] Place the air-dried soil in an evaporating dish and thoroughly mix with distilled water until the mass becomes plastic enough to be easily shaped into a ball. Take a portion of this ball weighing about 8 g for the test sample.

3.2 If both the liquid and plastic limits are required, take a test sample weighing about 8 g from the thoroughly wet and mixed portion of the soil prepared in accordance with ASTM Method D 423, Test for Liquid Limit of Soils.[2] Take the sample at any stage of the mixing process at which the mass becomes plastic enough to be easily shaped into a ball without sticking to the fingers excessively when squeezed. If the sample is taken before completion of the liquid limit test, set it aside and allow to season in air until the liquid limit test has been completed. If the sample is taken after completion of the liquid limit test, and is still too dry to permit rolling to a ⅛-in. (3.2-mm) thread, add more water.

4. Procedure

4.1 Squeeze and form the 8-g test sample taken in accordance with 3.1 or 3.2 into an ellipsoidal-shape mass. Roll this mass between the fingers and the ground-glass plate or a piece of paper lying on a smooth horizontal surface with just sufficient pressure to roll the mass into a thread of uniform diameter throughout its length. The rate of rolling shall be between 80 and 90 strokes/min.

[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock for Engineering Purposes.
Current edition approved Sept. 19, 1959. Originally issued 1935. Replaces D 424 - 54.

[2] *Annual Book of ASTM Standards*, Part 19.

counting a stroke as one complete motion of the hand forward and back to the starting position again.

4.2 When the diameter of the thread becomes ⅛ in. (3.2 mm), break the thread into six or eight pieces. Squeeze the pieces together between the thumbs and fingers of both hands into a uniform mass roughly ellipsoidal in shape, and reroll. Continue this alternate rolling to a thread ⅛ in. in diameter, gathering together, kneading and rerolling, until the thread crumbles under the pressure required for rolling and the soil can no longer be rolled into a thread. The crumbling may occur when the thread has a diameter greater than ⅛ in. This shall be considered a satisfactory end point, provided the soil has been previously rolled into a thread ⅛ in. in diameter. The crumbling will manifest itself differently with the various types of soil. Some soils fall apart in numerous small aggregations of particles; others may form an outside tubular layer that starts splitting at both ends. The splitting progresses toward the middle, and finally, the thread falls apart in many small platy particles. Heavy clay soils require much pressure to deform the thread, particularly as they approach the plastic limit, and finally, the thread breaks into a series of barrel-shaped segments each about ¼ to ⅜ in. (6.4 to 9.5 mm) in length. At no time shall the operator attempt to produce failure at exactly ⅛-in. diameter by allowing the thread to reach ⅛ in., then reducing the rate of rolling or the hand pressure, or both, and continuing the rolling without further deformation until the thread falls apart. It is permissible, however, to reduce the total amount of deformation for feebly plastic soils by making the initial diameter of the ellipsoidal-shaped mass nearer to the required ⅛ in. final diameter.

4.3 Gather the portions of the crumbled soil together and place in a suitable tared container. Weigh the container and soil and record the mass. Oven-dry the soil in the container to constant mass at 230 ± 9 F (110 ± 5 C) and weigh. Record this mass. Record the loss in mass as the mass of water.

5. Calculations

5.1 Calculate the plastic limit, expressed as the water content in percentage of the mass of the oven-dry soil, as follows:

Plastic limit
= (mass of water/mass of oven-dry soil) × 100

Report the plastic limit to the nearest whole number.

5.2 Calculate the plasticity index of a soil as the difference between its liquid limit and its plastic limit, as follows:

Plasticity index = liquid limit − plastic limit

5.3 Report the difference calculated as indicated in 5.2 as the plasticity index, except under the following conditions:

5.3.1 When the liquid limit or plastic limit cannot be determined, report the plasticity index as NP (nonplastic).

5.3.2 When the soil is extremely sandy, the plastic limit test shall be made before the liquid limit. If the plastic limit cannot be determined, report both the liquid limit and plastic limit as NP.

5.3.3 When the plastic limit is equal to, or greater than, the liquid limit, report the plasticity index as NP.

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AMERICAN NATIONAL STANDARD ANSI/ASTM D 422 - 63 (Reapproved 1972)

APPENDIX C-5

Standard Method for PARTICLE-SIZE ANALYSIS OF SOILS[1]

This Standard is issued under the fixed designation D 422; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval.

1. Scope

1.1 This method covers the quantitative determination of the distribution of particle sizes in soils. The distribution of particle sizes larger than 75 μm (retained on the No. 200 sieve) is determined by sieving, while the distribution of particle sizes smaller than 75 μm is determined by a sedimentation process, using a hydrometer to secure the necessary data (Notes 1 and 2).

NOTE 1—Separation may be made on the No. 4 (4.75-mm), No. 40 (425-μm), or No. 200 (75-μm) sieve instead of the No. 10. For whatever sieve used, the size shall be indicated in the report.

NOTE 2—Two types of dispersion devices are provided: (*1*) a high-speed mechanical stirrer, and (*2*) air dispersion. Extensive investigations indicate that air-dispersion devices produce a more positive dispersion of plastic soils below the 20-μm size and appreciably less degradation on all sizes when used with sandy soils. Because of the definite advantages favoring air dispersion, its use is recommended. The results from the two types of devices differ in magnitude, depending upon soil type, leading to marked differences in particle size distribution, especially for sizes finer than 20 μm.

2. Apparatus

2.1 *Balances*—A balance sensitive to 0.01 g for weighing the material passing a No. 10 (2.00-mm) sieve, and a balance sensitive to 0.1 percent of the mass of the sample to be weighed for weighing the material retained on a No. 10 sieve.

2.2 *Stirring Apparatus*—Either apparatus A or B may be used.

2.2.1 Apparatus A shall consist of a mechanically operated stirring device in which a suitably mounted electric motor turns a vertical shaft at a speed of not less than 10,000 rpm without load. The shaft shall be equipped with a replaceable stirring paddle made of metal, plastic, or hard rubber, as shown in Fig. 1. The shaft shall be of such length that the stirring paddle will operate not less than ¾ in. (19.0 mm) nor more than 1½ in. (38.1 mm) above the bottom of the dispersion cup. A special dispersion cup conforming to either of the designs shown in Fig. 2 shall be provided to hold the sample while it is being dispersed.

2.2.2 Apparatus B shall consist of an air-jet dispersion cup[2] (Note 3) conforming to the general details shown in Fig. 3 (Notes 4 and 5).

NOTE 3—The amount of air required by an air-jet dispersion cup is of the order of 2 ft³/min; some small air compressors are not capable of supplying sufficient air to operate a cup.

NOTE 4—Another air-type dispersion device, known as a dispersion tube, developed by Chu and Davidson at Iowa State College, has been shown to give results equivalent to those secured by the air-jet dispersion cups. When it is used, soaking of the sample can be done in the sedimentation cylinder, thus eliminating the need for transferring the slurry. When the air-dispersion tube is used, it shall be so indicated in the report.

NOTE 5—Water may condense in air lines when not in use. This water must be removed, either by using a water trap on the air line, or by blowing the water out of the line before using any of the air for dispersion purposes.

2.3 *Hydrometer*—An ASTM hydrometer, graduated to read in either specific gravity of the suspension or grams per litre of suspension, and conforming to the requirements for

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[1] This method is under the jurisdiction of ASTM Committee D-18 on Soil and Rock for Engineering Purposes.

Current edition accepted Nov. 21, 1963. Originally issued 1935. Replaces D 422 - 62.

[2] Detailed working drawings for this cup are available at a nominal cost from the American Society for Testing and Materials, 1916 Race St., Philadelphia, Pa. 19103. Order Adjunct No. 12-404220-00.

hydrometers 151H or 152H in ASTM Specification E 100, for ASTM Hydrometers.[3] Dimensions of both hydrometers are the same, the scale being the only item of difference.

2.4 *Sedimentation Cylinder*—A glass cylinder essentially 18 in. (457 mm) in height and 2½ in. (63.5 mm) in diameter, and marked for a volume of 1000 ml. The inside diameter shall be such that the 1000-ml mark is 36 ± 2 cm from the bottom on the inside.

2.5 *Thermometer*—A thermometer accurate to 1 F (0.5 C).

2.6 *Sieves*—A series of sieves, of square-mesh woven-wire cloth, conforming to the requirements of ASTM Specification E 11, for Wire-Cloth Sieves for Testing Purposes.[3] A full set of sieves includes the following (Note 6):

3-in. (75-mm)	No. 10 (2.00-mm)
2-in. (50-mm)	No. 20 (850-μm)
1½-in. (37.5-mm)	No. 40 (425-μm)
1-in. (25.0-mm)	No. 60 (250-μm)
¾-in. (19.0-mm)	No. 140 (106-μm)
⅜-in. (9.5-mm)	No. 200 (75-μm)
No. 4 (4.75-mm)	

NOTE 6—A set of sieves giving uniform spacing of points for the graph, as required in Section 16, may be used if desired. This set consists of the following sieves:

3-in. (75-mm)	No. 16 (1.18-mm)
1½-in. (37.5-mm)	No. 30 (600-μm)
¾-in. (19.0-mm)	No. 50 (300-μm)
⅜-in. (9.5-mm)	No. 100 (150-μm)
No. 4 (4.75-mm)	No. 200 (75-μm)
No. 8 (2.36-mm)	

2.7 *Water Bath or Constant-Temperature Room*—A water bath or constant-temperature room for maintaining the soil suspension at a constant temperature during the hydrometer analysis. A satisfactory water tank is an insulated tank that maintains the temperature of the suspension at a convenient constant temperature at or near 68 F (20 C). Such a device is illustrated in Fig. 4. In cases where the work is performed in a room at an automatically controlled constant temperature, the water bath is not necessary.

2.8 *Beaker*—A beaker of 250-ml capacity.

2.9 *Timing Device*—A watch or clock with a second hand.

3. Dispersing Agent

3.1 A solution of sodium hexametaphosphate (sometimes called sodium metaphosphate) shall be used in distilled or demineralized water, at the rate of 40 g of sodium hexametaphosphate/litre of solution (Note 7).

NOTE 7—Solutions of this salt, if acidic, slowly revert or hydrolyze back to the orthophosphate form with a resultant decrease in dispersive action. Solutions should be prepared frequently (at least once a month) or adjusted to pH of 8 or 9 by means of sodium carbonate. Bottles containing solutions should have the date of preparation marked on them.

3.2 All water used shall be either distilled or demineralized water. The water for a hydrometer test shall be brought to the temperature that is expected to prevail during the hydrometer test. For example, if the sedimentation cylinder is to be placed in the water bath, the distilled or demineralized water to be used shall be brought to the temperature of the controlled water bath; or, if the sedimentation cylinder is used in a room with controlled temperature, the water for the test shall be at the temperature of the room. The basic temperature for the hydrometer test is 68 F (20 C). Small variations of temperature do not introduce differences that are of practical significance and do not prevent the use of corrections derived as prescribed.

4. Test Sample

4.1 Prepare the test sample for mechanical analysis as outlined in ASTM Method D 421, Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constants.[4] During the preparation procedure the sample is divided into two portions. One portion contains only particles retained on the No. 10 (2.00-mm) sieve while the other portion contains only particles passing the No. 10 sieve. The mass of air-dried soil selected for purpose of tests, as prescribed in Method D 421, shall be sufficient to yield quantities for mechanical analysis as follows:

4.1.1 The size of the portion retained on the No. 10 sieve shall depend on the maximum size of particle, according to the following schedule:

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
⅜ (9.5)	500
¾ (19.0)	1000

[3] *Annual Book of ASTM Standards*, Part 41.
[4] *Annual Book of ASTM Standards*, Part 19.

Nominal Diameter of Largest Particles, in. (mm)	Approximate Minimum Mass of Portion, g
1 (25.4)	2000
1½ (38.1)	3000
2 (50.8)	4000
3 (76.2)	5000

4.1.2 The size of the portion passing the No. 10 sieve shall be approximately 115 g for sandy soils and approximately 65 g for silt and clay soils.

4.2 Provision is made in Section 4 of Method D 421 for the weighing of the air-dry soil selected for purpose of tests, the separation of the soil on the No. 10 sieve by dry-sieving and washing, and the weighing of the washed and dried fraction retained on the No. 10 sieve. From these two masses the percentages retained and passing the No. 10 sieve can be calculated in accordance with 11.1.

NOTE 8—A check on the mass values and the thoroughness of pulverization of the clods may be secured by weighing the portion passing the No. 10 sieve and adding this value to the mass of the washed and oven-dried portion retained on the No. 10 sieve.

SIEVE ANALYSIS OF PORTION RETAINED ON NO. 10 (2.00-mm) SIEVE

5. Procedure

5.1 Separate the portion retained on the No. 10 (2.00-mm) sieve into a series of fractions using the 3-in. (75-mm), 2-in. (50-mm), 1½-in. (37.5-mm), 1-in. (25.0-mm), ¾-in. (19.0-mm), ⅜-in. (9.5-mm), No. 4 (4.75-mm), and No. 10 sieves, or as many as may be needed depending on the sample, or upon the specifications for the material under test.

5.2 Conduct the sieving operation by means of a lateral and vertical motion of the sieve, accompanied by a jarring action in order to keep the sample moving continuously over the surface of the sieve. In no case turn or manipulate fragments in the sample through the sieve by hand. Continue sieving until not more than 1 mass percent of the residue on a sieve passes that sieve during 1 min of sieving. When mechanical sieving is used, test the thoroughness of sieving by using the hand method of sieving as described above.

5.3 Determine the mass of each fraction on a balance conforming to the requirements of 2.1. At the end of weighing, the sum of the masses retained on all the sieves used should equal closely the original mass of the quantity sieved.

HYDROMETER AND SIEVE ANALYSIS OF PORTION PASSING THE NO. 10 (2.00-mm) SIEVE

6. Determination of Composite Correction for Hydrometer Reading

6.1 Equations for percentages of soil remaining in suspension, as given in 13.3, are based on the use of distilled or demineralized water. A dispersing agent is used in the water, however, and the specific gravity of the resulting liquid is appreciably greater than that of distilled or demineralized water.

6.1.1 Both soil hydrometers are calibrated at 68 F (20 C), and variations in temperature from this standard temperature produce inaccuracies in the actual hydrometer readings. The amount of the inaccuracy increases as the variation from the standard temperature increases.

6.1.2 Hydrometers are graduated by the manufacturer to be read at the bottom of the meniscus formed by the liquid on the stem. Since it is not possible to secure readings of soil suspensions at the bottom of the meniscus, readings must be taken at the top and a correction applied.

6.1.3 The net amount of the corrections for the three items enumerated is designated as the composite correction, and may be determined experimentally.

6.2 For convenience, a graph or table of composite corrections for a series of 1-deg temperature differences for the range of expected test temperatures may be prepared and used as needed. Measurement of the composite corrections may be made at two temperatures spanning the range of expected test temperatures, and corrections for the intermediate temperatures calculated assuming a straight-line relationship between the two observed values.

6.3 Prepare 1000 ml of liquid composed of distilled or demineralized water and dispersing agent in the same proportion as will prevail in the sedimentation (hydrometer) test. Place the liquid in a sedimentation cylinder and the cylinder in the constant-temperature water bath, set for one of the two temperatures to be used. When the tempera-

ture of the liquid becomes constant, insert the hydrometer, and, after a short interval to permit the hydrometer to come to the temperature of the liquid, read the hydrometer at the top of the meniscus formed on the stem. For hydrometer 151H the composite correction is the difference between this reading and one: for hydrometer 152H it is the difference between the reading and zero. Bring the liquid and the hydrometer to the other temperature to be used, and secure the composite correction as before.

7. Hygroscopic Moisture

7.1 When the sample is weighed for the hydrometer test, weigh out an auxiliary portion of from 10 to 15 g in a small metal or glass container, dry the sample to a constant mass in an oven at 230 ± 9 F (110 ± 5 C), and weigh again. Record the masses.

8. Dispersion of Soil Sample

8.1 When the soil is mostly of the clay and silt sizes, weigh out a sample of air-dry soil of approximately 50 g. When the soil is mostly sand the sample should be approximately 100 g.

8.2 Place the sample in the 250-ml beaker and cover with 125 ml of sodium hexametaphosphate solution (40 g/litre). Stir until the soil is thoroughly wetted. Allow to soak for at least 16 h.

8.3 At the end of the soaking period, disperse the sample further, using either stirring apparatus A or B. If stirring apparatus A is used, transfer the soil - water slurry from the beaker into the special dispersion cup shown in Fig. 2, washing any residue from the beaker into the cup with distilled or demineralized water (Note 9). Add distilled or demineralized water, if necessary, so that the cup is more than half full. Stir for a period of 1 min.

NOTE 9—A large size syringe is a convenient device for handling the water in the washing operation. Other devices include the wash-water bottle and a hose with nozzle connected to a pressurized distilled water tank.

8.4 If stirring apparatus B (Fig. 3) is used, remove the cover cap and connect the cup to a compressed air supply by means of a rubber hose. An air gage must be on the line between the cup and the control valve. Open the control valve so that the gage indicates 1 psi (7 kPa) pressure (Note 10). Transfer the soil-water slurry from the beaker to the air-jet dispersion cup by washing with distilled or demineralized water. Add distilled or demineralized water, if necessary, so that the total volume in the cup is 250 ml, but no more.

NOTE 10—The initial air pressure of 1 psi is required to prevent the soil - water mixture from entering the air-jet chamber when the mixture is transferred to the dispersion cup.

8.5 Place the cover cap on the cup and open the air control valve until the gage pressure is 20 psi (140 kPa). Disperse the soil according to the following schedule:

Plasticity Index	Dispersion Period, min
Under 5	5
6 to 20	10
Over 20	15

Soils containing large percentages of mica need be dispersed for only 1 min. After the dispersion period, reduce the gage pressure to 1 psi preparatory to transfer of soil - water slurry to the sedimentation cylinder.

9. Hydrometer Test

9.1 Immediately after dispersion, transfer the soil - water slurry to the glass sedimentation cylinder, and add distilled or demineralized water until the total volume is 1000 ml.

9.2 Using the palm of the hand over the open end of the cylinder (or a rubber stopper in the open end), turn the cylinder upside down and back for a period of 1 min to complete the agitation of the slurry (Note 11). At the end of 1 min set the cylinder in a convenient location and take hydrometer readings at the following intervals of time (measured from the beginning of sedimentation), or as many as may be needed, depending on the sample or the specification for the material under test: 2, 5, 15, 30, 60, 250, and 1440 min. If the controlled water bath is used, the sedimentation cylinder should be placed in the bath between the 2- and 5-min readings.

NOTE 11—The number of turns during this minute should be approximately 60, counting the turn upside down and back as two turns. Any soil remaining in the bottom of the cylinder during the first few turns should be loosened by vigorous shaking of the cylinder while it is in the inverted position.

9.3 When it is desired to take a hydrometer reading, carefully insert the hydrometer about

20 to 25 s before the reading is due to approximately the depth it will have when the reading is taken. As soon as the reading is taken, carefully remove the hydrometer and place it with a spinning motion in a graduate of clean distilled or demineralized water.

NOTE 12—It is important to remove the hydrometer immediately after each reading. Readings shall be taken at the top of the meniscus formed by the suspension around the stem, since it is not possible to secure readings at the bottom of the meniscus.

9.4 After each reading, take the temperature of the suspension by inserting the thermometer into the suspension.

10. Sieve Analysis

10.1 After taking the final hydrometer reading, transfer the suspension to a No. 200 (75-μm) sieve and wash with tap water until the wash water is clear. Transfer the material on the No. 200 sieve to a suitable container, dry in an oven at 230 ± 9 F (110 ± 5 C) and make a sieve analysis of the portion retained, using as many sieves as desired, or required for the material, or upon the specification of the material under test.

CALCULATIONS AND REPORT

11. Sieve Analysis Values for the Portion Coarser than the No. 10 (2.00-mm) Sieve

11.1 Calculate the percentage passing the No. 10 sieve by dividing the mass passing the No. 10 sieve by the mass of soil originally split on the No. 10 sieve, and multiplying the result by 100. To obtain the mass passing the No. 10 sieve, subtract the mass retained on the No. 10 sieve from the original mass.

11.2 To secure the total mass of soil passing the No. 4 (4.75-mm) sieve, add to the mass of the material passing the No. 10 sieve the mass of the fraction passing the No. 4 sieve and retained on the No. 10 sieve. To secure the total mass of soil passing the ⅜-in. (9.5-mm) sieve, add to the total mass of soil passing the No. 4 sieve, the mass of the fraction passing the ⅜-in. sieve and retained on the No. 4 sieve. For the remaining sieves, continue the calculations in the same manner.

11.3 To determine the total percentage passing for each sieve, divide the total mass passing (see 11.2) by the total mass of sample and multiply the result by 100.

12. Hygroscopic Moisture Correction Factor

12.1 The hygroscopic moisture correction factor is the ratio between the mass of the oven-dried sample and the air-dry mass before drying. It is a number less than one, except when there is no hygroscopic moisture.

13. Percentages of Soil in Suspension

13.1 Calculate the oven-dry mass of soil used in the hydrometer analysis by multiplying the air-dry mass by the hygroscopic moisture correction factor.

13.2 Calculate the mass of a total sample represented by the mass of soil used in the hydrometer test, by dividing the oven-dry mass used by the percentage passing the No. 10 (2.00-mm) sieve, and multiplying the result by 100. This value is the weight W in the equation for percentage remaining in suspension.

13.3 The percentage of soil remaining in suspension at the level at which the hydrometer is measuring the density of the suspension may be calculated as follows (Note 13):
For hydrometer 151H:

$$P = [(100.000/W) \times G/(G - G_1)] (R - G_1)$$

NOTE 13—The bracketed portion of the equation for hydrometer 151H is constant for a series of readings and may be calculated first and then multiplied by the portion in the parenthesis.

For hydrometer 152H:

$$P = (Ra/W) \times 100$$

where:

a = correction faction to be applied to the reading of hydrometer 152H. (Values shown on the scale are computed using a specific gravity of 2.65. Correction factors are given in Table 1),
P = percentage of soil remaining in suspension at the level at which the hydrometer measures the density of the suspension,
R = hydrometer reading with composite correction applied (Section 6),
W = oven-dry mass of soil in a total test sample represented by mass of soil dispersed (see 13.2), g,
G = specific gravity of the soil particles, and
G_1 = specific gravity of the liquid in which soil particles are suspended. Use nu-

merical value of one in both instances in the equation. In the first instance any possible variation produces no significant effect, and in the second instance, the composite correction for R is based on a value of one for G_1.

Diameter of Soil Particles

14.1 The diameter of a particle corresponding to the percentage indicated by a given hydrometer reading shall be calculated according to Stokes' law (Note 14), on the basis that a particle of this diameter was at the surface of the suspension at the beginning of sedimentation and had settled to the level at which the hydrometer is measuring the density of the suspension. According to Stokes' law:

$$D = \sqrt{[30n/980(G - G_1)] \times L/T}$$

where:

D = diameter of particle, mm,
n = coefficient of viscosity of the suspending medium (in this case water) in poises (varies with changes in temperature of the suspending medium),
L = distance from the surface of the suspension to the level at which the density of the suspension is being measured, cm. (For a given hydrometer and sedimentation cylinder, values vary according to the hydrometer readings. This distance is known as effective depth (Table 2),
T = interval of time from beginning of sedimentation to the taking of the reading, min,
G = specific gravity of soil particles, and
G_1 = specific gravity (relative density) of suspending medium (value may be used as 1.000 for all practical purposes).

NOTE 14—Since Stokes' law considers the terminal velocity of a single sphere falling in an infinity of liquid, the sizes calculated represent the diameter of spheres that would fall at the same rate as the soil particles.

14.2 For convenience in calculations the above equation may be written as follows:

$$D = K\sqrt{L/T}$$

where:

K = constant depending on the temperature of the suspension and the specific gravity of the soil particles. Values of K for a range of temperatures and specific gravities are given in Table 3. The value of K does not change for a series of readings constituting a test, while values of L and T do vary.

14.3 Values of D may be computed with sufficient accuracy, using an ordinary 10-in. slide rule.

NOTE 15—The value of L is divided by T using the A- and B-scales, the square root being indicated on the D-scale. Without ascertaining the value of the square root it may be multiplied by K, using either the C- or CI-scale.

15. Sieve Analysis Values for Portion Finer than No. 10 (2.00-mm) Sieve

15.1 Calculation of percentages passing the various sieves used in sieving the portion of the sample from the hydrometer test involves several steps. The first step is to calculate the mass of the fraction that would have been retained on the No. 10 sieve had it not been removed. This mass is equal to the total percentage retained on the No. 10 sieve (100 minus total percentage passing) times the mass of the total sample represented by the mass of soil used (as calculated in 13.2), and the result divided by 100.

15.2 Calculate next the total mass passing the No. 200 sieve. Add together the fractional masses retained on all the sieve including the No. 10 sieve, and subtract this sum from the mass of the total sample (as calculated in 13.2).

15.3 Calculate next the total masses passing each of the other sieves, in a manner similar to that given in 11.2.

15.4 Calculate last the total percentages passing by dividing the total mass passing (as calculated in 15.3) by the total mass of sample (as calculated in 13.2), and multiply the result by 100.

16. Graph

16.1 When the hydrometer analysis is performed, a graph of the test results shall be made, plotting the diameters of the particles on a logarithmic scale as the abscissa and the percentages smaller than the corresponding diameters to an arithmetic scale as the ordinate. When the hydrometer analysis is not

made on a portion of the soil, the preparation of the graph is optional, since values may be secured directly from tabulated data.

17. Report

17.1 The report shall include the following:

17.1.1 Maximum size of particles,

17.1.2 Percentage passing (or retained on) each sieve, which may be tabulated or presented by plotting on a graph (Note 16),

17.1.3 Description of sand and gravel particles:

17.1.3.1 Shape—rounded or angular,

17.1.3.2 Hardness—hard and durable, soft, or weathered and friable,

17.1.4 Specific gravity, if unusually high or low,

17.1.5 Any difficulty in dispersing the fraction passing the No. 10 (2.00-mm) sieve, indicating any change in type and amount of dispersing agent, and

17.1.6 The dispersion device used and the length of the dispersion period.

NOTE 16—This tabulation of graph represents the gradation of the sample tested. If particles larger than those contained in the sample were removed before testing, the report shall so state giving the amount and maximum size.

17.2 For materials tested for compliance with definite specifications, the fractions called for in such specifications shall be reported. The fractions smaller than the No. 10 sieve shall be read from the graph.

17.3 For materials for which compliance with definite specifications is not indicated and when the soil is composed almost entirely of particles passing the No. 4 (4.75-mm) sieve, the results read from the graph may be reported as follows:

(*1*) Gravel, passing 3-in. and retained on No. 4 sieve ..... percent
(*2*) Sand, passing No. 4 sieve and retained on No. 200 sieve ..... percent
(*a*) Coarse sand, passing No. 4 sieve and retained on No. 10 sieve ..... percent
(*b*) Medium sand, passing No. 10 sieve and retained on No. 40 sieve ..... percent
(*c*) Fine sand, passing No. 40 sieve and retained on No. 200 sieve ..... percent
(*3*) Silt size, 0.074 to 0.005 mm ..... percent
(*4*) Clay size, smaller than 0.005 mm ..... percent
Colloids, smaller than 0.001 mm ..... percent

17.4 For materials for which compliance with definite specifications is not indicated and when the soil contains material retained on the No. 4 sieve sufficient to require a sieve analysis on that portion, the results may be reported as follows (Note 17):

SIEVE ANALYSIS

Sieve Size	Percentage Passing
3-in.	.....
2-in.	.....
1½-in.	.....
1-in.	.....
¾-in.	.....
⅜-in.	.....
No. 4 (4.75-mm)	.....
No. 10 (2.00-mm)	.....
No. 40 (425-μm)	.....
No. 200 (75-μm)	.....

HYDROMETER ANALYSIS

0.074 mm	.....
0.005 mm	.....
0.001 mm	.....

NOTE 17—No. 8 (2.36-mm) and No. 50 (300-μm) sieves may be substituted for No. 10 and No. 40 sieves.

TABLE 1 Values of Correction Factor, *a*, for Different Specific Gravities of Soil Particles[a]

Specific Gravity	Correction Factor[a]
2.95	0.94
2.90	0.95
2.85	0.96
2.80	0.97
2.75	0.98
2.70	0.99
2.65	1.00
2.60	1.01
2.55	1.02
2.50	1.03
2.45	1.05

[a] For use in equation for percentage of soil remaining in suspension when using Hydrometer 152H.

TABLE 2 Values of Effective Depth Based on Hydrometer and Sedimentation Cylinder of Specified Sizes[a]

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, *L*, cm	Actual Hydrometer Reading	Effective Depth, *L*, cm	Actual Hydrometer Reading	Effective Depth, *L*, cm
1.000	16.3	0	16.3	31	11.2
1.001	16.0	1	16.1	32	11.1
1.002	15.8	2	16.0	33	10.9
1.003	15.5	3	15.8	34	10.7
1.004	15.2	4	15.6	35	10.6
1.005	15.0	5	15.5		
1.006	14.7	6	15.3	36	10.4
1.007	14.4	7	15.2	37	10.2
1.008	14.2	8	15.0	38	10.1
1.009	13.9	9	14.8	39	9.9
1.010	13.7	10	14.7	40	9.7
1.011	13.4	11	14.5	41	9.6
1.012	13.1	12	14.3	42	9.4
1.013	12.9	13	14.2	43	9.2
1.014	12.6	14	14.0	44	9.1
1.015	12.3	15	13.8	45	8.9
1.016	12.1	16	13.7	46	8.8
1.017	11.8	17	13.5	47	8.6
1.018	11.5	18	13.3	48	8.4
1.019	11.3	19	13.2	49	8.3
1.020	11.0	20	13.0	50	8.1
1.021	10.7	21	12.9	51	7.9
1.022	10.5	22	12.7	52	7.8
1.023	10.2	23	12.5	53	7.6
1.024	10.0	24	12.4	54	7.4
1.025	9.7	25	12.2	55	7.3
1.026	9.4	26	12.0	56	7.1
1.027	9.2	27	11.9	57	7.0
1.028	8.9	28	11.7	58	6.8
1.029	8.6	29	11.5	59	6.6
1.030	8.4	30	11.4	60	6.5

Table 2 *Continued*

Hydrometer 151H		Hydrometer 152H			
Actual Hydrometer Reading	Effective Depth, *L*, cm	Actual Hydrometer Reading	Effective Depth, *L*, cm	Actual Hydrometer Reading	Effective Depth, *L*, cm
1.031	8.1				
1.032	7.8				
1.033	7.6				
1.034	7.3				
1.035	7.0				
1.036	6.8				
1.037	6.5				
1.038	6.2				

[a] Values of effective depth are calculated from the equation:

$$L = L_1 + \tfrac{1}{2}\,[L_2 - (V_B/A)]$$

where:

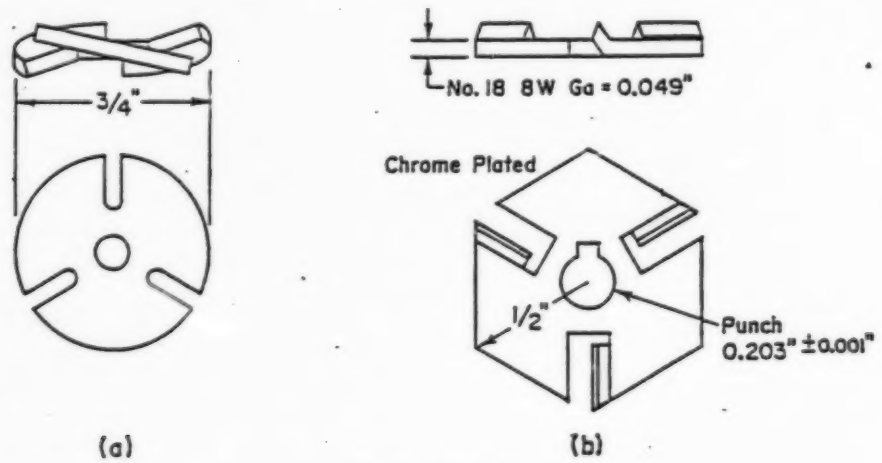
L = effective depth, cm,
L_1 = distance along the stem of the hydrometer from the top of the bulb to the mark for a hydrometer reading, cm,
L_2 = overall length of the hydrometer bulb, cm,
V_B = volume of hydrometer bulb, cm^3, and
A = cross-sectional area of sedimentation cylinder, cm^2.

Values used in calculating the values in Table 2 are as follows:

For both hydrometers, 151H and 152H:
L_2 = 14.0 cm
V_B = 67.0 cm^3
A = 27.8 cm^2
For hydrometer 151H:
L_1 = 10.5 cm for a reading of 1.000
= 2.3 cm for a reading of 1.031
For hydrometer 152H:
L_1 = 10.5 cm for a reading of 0 g/litre
= 2.3 cm for a reading of 50 g/litre

TABLE 3 Values of K for Use in Equation for Computing Diameter of Particle in Hydrometer Analysis

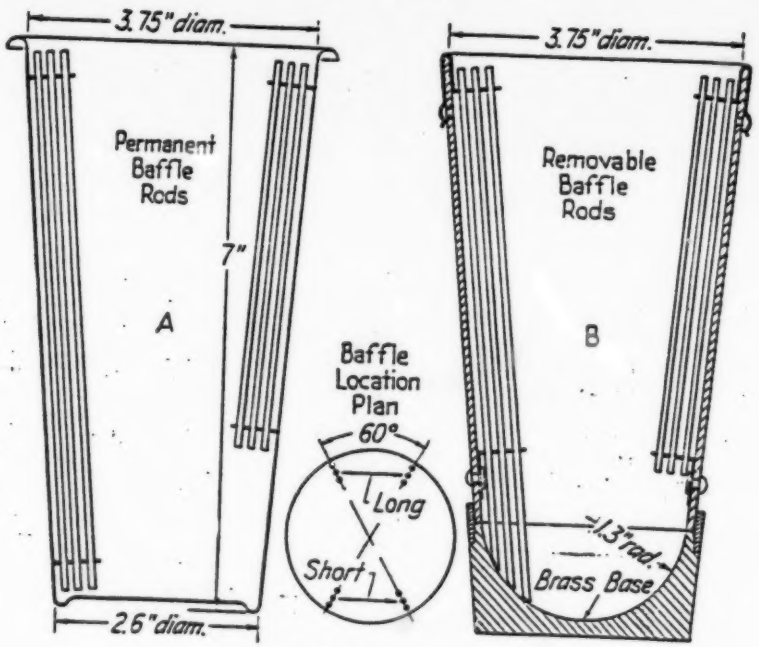
Temperature, deg C	Specific Gravity of Soil Particles								
	2.45	2.50	2.55	2.60	2.65	2.70	2.75	2.80	2.85
16	0.01510	0.01505	0.01481	0.01457	0.01435	0.01414	0.01394	0.01374	0.01356
17	0.01511	0.01486	0.01462	0.01439	0.01417	0.01396	0.01376	0.01356	0.01338
18	0.01492	0.01467	0.01443	0.01421	0.01399	0.01378	0.01359	0.01339	0.01321
19	0.01474	0.01449	0.01425	0.01403	0.01382	0.01361	0.01342	0.01323	0.01305
20	0.01456	0.01431	0.01408	0.01386	0.01365	0.01344	0.01325	0.01307	0.01289
21	0.01438	0.01414	0.01391	0.01369	0.01348	0.01328	0.01309	0.01291	0.01273
22	0.01421	0.01397	0.01374	0.01353	0.01332	0.01312	0.01294	0.01276	0.01258
23	0.01404	0.01381	0.01358	0.01337	0.01317	0.01297	0.01279	0.01261	0.01243
24	0.01388	0.01365	0.01342	0.01321	0.01301	0.01282	0.01264	0.01246	0.01229
25	0.01372	0.01349	0.01327	0.01306	0.01286	0.01267	0.01249	0.01232	0.01215
26	0.01357	0.01334	0.01312	0.01291	0.01272	0.01253	0.01235	0.01218	0.01201
27	0.01342	0.01319	0.01297	0.01277	0.01258	0.01239	0.01221	0.01204	0.01188
28	0.01327	0.01304	0.01283	0.01264	0.01244	0.01225	0.01208	0.01191	0.01175
29	0.01312	0.01290	0.01269	0.01249	0.01230	0.01212	0.01195	0.01178	0.01162
30	0.01298	0.01276	0.01256	0.01236	0.01217	0.01199	0.01182	0.01165	0.01149



Metric Equivalents

in.	0.00?	0.049	0.203	½	¾
mm	0.03	1.24	5.16	12.7	19.0

FIG. 1 Detail of Stirring Paddles.



Metric Equivalents

in.	1.3	2.6	3.75
mm	33	66	95.2

FIG. 2 Dispersion Cups of Apparatus A.

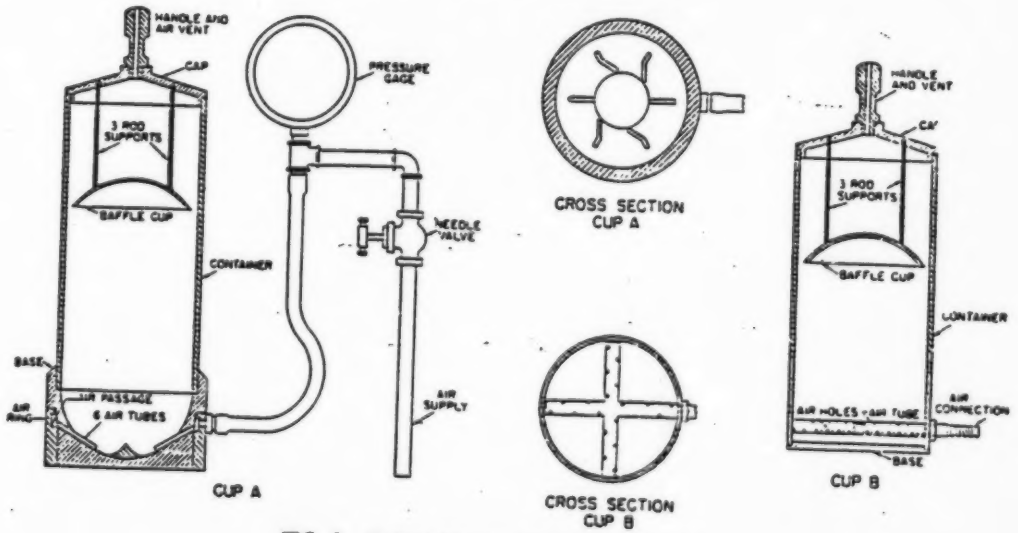
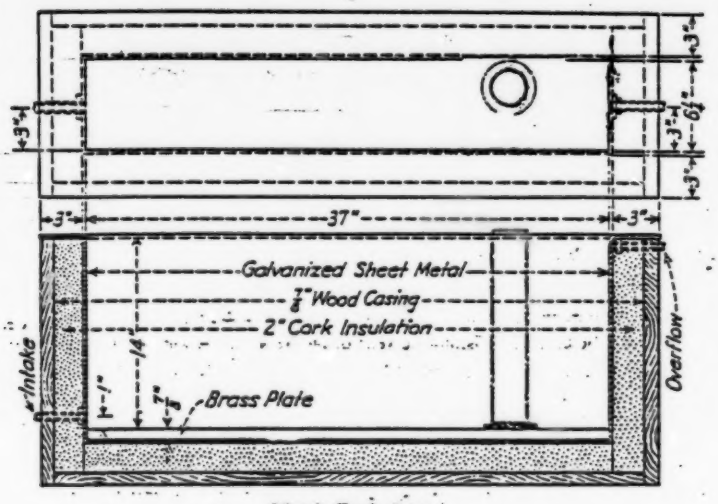


FIG. 3 Air-Jet Dispersion Cups of Apparatus B.



Metric Equivalents

in.	⅛	1	3	6¼	14	37
mm	22.2	25.4	76.2	158.2	356	940

FIG. 4 Insulated Water Bath.

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APPENDIX C-6

DETERMINATION OF SLAKE-DURABILITY INDEX

SCOPE

41. a. The purpose of this test is to assess the rock sample's resistance to weakening and disintegration when subjected to two standard cycles of drying and wetting.

b. All procedures for determining the slake-durability index are based on the appropriate standards (2).

APPARATUS

42. The apparatus consists essentially of the following:

a. A test drum, comprising a 2.00 mm mesh cylinder of unobstructed length 100 mm and of diameter 140 mm, fixed to a solid base. The drum has a solid removable lid held in place by three bolts located between the base and the lid. The mesh cylinder must be sufficiently strong to retain its shape during use but neither its exterior or interior should be obstructed, for example, by reinforcing members. The drum must withstand a temperature of 105° C.

b. A trough to contain the test drum supported horizontally by its axis to allow free rotation, and with water capacity to a level of 20 mm below the drum axis. The drum is mounted to allow 40 mm unobstructed clearance between the mesh cylinder and trough base. The principal features of the trough and drum assembly are illustrated in Fig 6.

c. A motor drive capable of rotating the drum at a speed of 20 rpm, speed to be held constant to within five per cent for a period of 10 min.

d. An oven with constant temperature of 105° C to within 3° C for a period of at least 12 hrs.

e. A balance capable of weighing the drum plus sample to an accuracy of 0.5 g.

PROCEDURE

43. a. A representative sample is selected comprising 10 rock lumps, each weighing about 50 g. The lumps should be spheroidal in shape and the corners should be rounded during preparation.

b. The sample is placed in the clean drum and dried to a constant weight at a temperature of 105° C, usually requiring from two to six hrs in the oven. The weight, A, of the drum together with the sample is recorded. The sample is then immediately tested.

c. The lid is replaced and the drum mounted in the trough and coupled to the motor.

d. The trough is filled with tap water at 20° C to a level 20 mm below the drum axis and the drum is rotated at 20 rpm for 10 min.

e. The drum is removed from the trough, the lid removed from the drum and the drum with

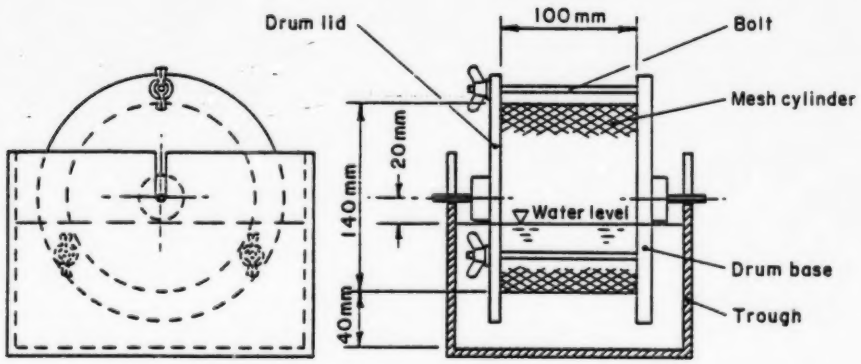

Fig 6 - Slake-durability test equipment.

retained portion of the sample dried to a constant weight at 105° C. The weight, B, of the drum including that of the retained portion of the sample is recorded.

f. Steps (c) - (e) are repeated and weight, C, of the drum including that of the retained portion of the sample is recorded.

g. The drum is cleaned and its weight, D, recorded.

CALCULATIONS

44. The slake-durability index (second cycle) is calculated as the percentage ratio of the final to initial dry sample weights as follows:

$$\text{Slake durability index, } I_{d2} = \frac{C-D}{A-D} \times 100\%$$

REPORTING OF RESULTS

45. The report should include the following information for each sample tested:

a. sample identification (rock type and origin).

b. slake-durability index (second cycle) to the nearest 0.1 per cent

c. general appearance of fragments retained in the drum

d. general appearance of material that has passed through the drum

e. nature and temperature of slaking fluid; this is usually tap water at 20° C, but could also be natural groundwater, sea water, etc.

46. The working sheets substitute for a report (Fig 7) if only a few sample locations are involved in determining the slake-durability index.

SLAKE-DURABILITY INDEX

Sample *shale of the hanging-wall contact zone* Date *July 4, 1972*

Tested by *W.G.*

Location *Section 21, second bench*

Bore Hole _____ Depth _____

Sample No. *H-S-3*

Weight A = *1263.0* g Weight C = *962.5* g

Weight B = *1179.5* g Weight D = *742.0* g

Slake-durability index, $I_{d2} = \frac{C-D}{A-D} \times 100\% = \frac{962.5-742.0}{1263.0-742.0} 100 =$ *42.3* %

Remarks: *approx. 3 mm thick wafer-like fragments in the drum and flaky fragments in the water, tap water at 19°C.*

Fig 7 - Working sheet for slake-durability index determination.

APPENDIX C-7

Cuesta Research Ltd May 2, 1986

X-ray diffraction analyses

SEATEARTHS

for Jacques Whitford and Associates Ltd

Four X-ray diffractograms were produced on clay samples provided by Baruck Wile of Jacques Whitford and Associates in April of 1986.

The samples were prepared by grinding in a hand mortar in acetone, spreading the resulting slurry on a glass slide and allowing the sample to dry. This produces an sample mount that is a uniform thickness of grains and in which the individual grains are relatively unoriented.

The samples were then run in a Phillips X-ray diffractometer using a copper lamp (wavelength = 1.54050 angstroms) and a power of 40 kv. The resultant energy was recorded on a chart recorder. The runs were all at a rate of one degree per minute at a chart speed of one centimetre per minute (thus one degree per minute). Degrees are measures of 2-theta which can be converted to D-spacing by using Bragg's equation. D-spacings can then be assigned to appropriate minerals by consulting any of several standard search manuels.

Runs were begun at 2-theta = 3 degrees to assure recording of low angle peaks typical of clay minerals. A salt standard was

included in all runs: the salt was NaCl and the peaks were clearly recorded on all runs indicating good resolution and little drift or chart irregularities.

D-spacing and mineralogy interpretation are provided on the chart recordings attached. In summary, the two samples from Lingan (Lingan 9E and Lingan 6W) are similar. They show strong and well defined illite and kaolinite peaks. They also show quartz peaks indicating a silt content of quartz. No feldspar peaks are evident. A low angle peak may be Fe-rich chlorite.

The Evans sample is similar to the Lingan samples in having dominant illite and kaolinite peaks with quartz. There is however, no evidence of chlorite. There are clearly defined but subordinant peaks for pyrite. Low angle peaks for illite are difuse perhaps indicating some disorder or some other interfering mineral. Montmorillanite peaks are however absent. Montmorillanite peaks can clearly be seen in the low angle area of the standard sample of the Bearpaw ~~coal~~ shale for comparison.

No clear montmorillanite or smectite peaks were found in the Nova Scotia samples.

Feldspar and calcite peaks are not a significant component of the Nova Scotia samples. This should prove useful in evaluating lithochemical data. It would appear that most of the

K, Na, and Ca must be in the clay minerals in these samples.

Please consult the chart recordings of the individual samples for peak details. Machine settings for each run are also recorded on these charts. Relative peak importance can be qualitatively estimated by comparing the area under the peaks. The dominant peak in all diagrams is an interference peak of both quartz and illite. Quantitative information should not be inferred from these diffractograms: major components will show dominant and clearly defined peaks; trace components will be low area peaks with low clarity. Significant trace component peaks can be masked by dominant peaks, though no concealed peaks would be expected in these analyses.

Further information and analyses are available from Cuesta Research Ltd.

APPENDIX C-8

ADSORPTION ISOTHERM TEST

SOURCE: Dennis E. O'Brien and Martin E. Chenevert, Stabilization of Sensitive Shales Using Inhibited Potasium-Based Drilling Fluids. Journal of Petroleum Technology September 1973, page 1092.

Adsorption Isotherms

Adsorption isotherm tests are useful for determining the amount of water a particular shale would contain under various equilibrium conditions. With this information one can estimate how much swelling will occur in the formation. The difference between the in-situ water content and the equilibrium value measured by the test represents a potential hydrational swelling limit.

The water content of the shale is measured as a function of water activity or relative humidity defined as

$$a = p/p_o \ , \quad . \ . \ . \ . \ . \ . \ . \ . \ . \ . \ . \quad (1)$$

where a is the water activity, p is the vapor pressure in contact with the shale, and p_o is the vapor pressure of pure water at the same temperature. A range of water activities is achieved using a number of saturated salt solutions (Table 3) placed in desiccators. Water contents measured at various water activities can be plotted vs p/p_o to yield an adsorption isotherm.[1]

Normally, the use of the adsorption isotherm involves estimating the in-situ water content for nonpreserved cores. A method of determining this value has been described previously.[1] By entering the isotherm at the in-situ estimate one can determine the equilibrium water activity. In general, the lower this value the more prone to sloughing the shale will be. The isotherm also gives indirect evidence of how much expandable clay — particularly montmorillonite—is present. Combining isotherm data with X-ray diffraction results (Table 4) shows that the shales with higher montmorillonite content in the first and second class adsorb the most water and that the adsorption decreases as the classification number increases.

TABLE 3—WATER ACTIVITIES OF VARIOUS SATURATED SALT SOLUTIONS

Desiccator Number	Salt Employed	p/p_o
1	$ZnCl_2$	0.100
2	$CaCl_2$	0.295
3	$Ca(NO_3)_2$	0.505
4	NaCl	0.755
5	$(NH_4)_2SO_4$	0.800
6	$Na_2C_4H_4O_6 \cdot 2H_2O$	0.920
7	KH_2PO_4	0.960
8	$K_2Cr_2O_7$	0.980

TABLE 4—COMPARISON OF X-PAY ANALYSES WITH ADSORPTION ISOTHERM DATA

Shale	Class Determined by X-Ray Analysis	Range of Percent Water Content at $p/p_o = 0.50$
Anahauc	1	> 4.0
Midway	3	2.4 to 2.8
Wolfcamp	4	1.5 to 2.0
Canadian Hard Shale	5	0.4 to 1.0